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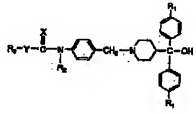
(54) NEW PIPERIDINE DERIVATIVE AND INSECTICIDE CONTAINING THE SAME

(57) Abstract:

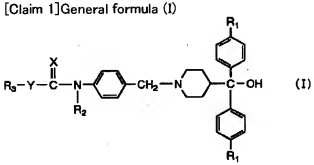
PROBLEM TO BE SOLVED: To provide piperidine derivative having a significantly excellent insecticidal activity with low dosage, compared with a conventional piperidine compound.

SOLUTION: This invention relates to an insecticide, acaricide or nematicide containing piepridine derivative represented by the general formula (1), N-oxide or a salt of the same and each of them as an effective component. [wherein, two R1s may be the same or different, respectively hydrogen atom, halogen atom, lower alkyl, lower alkoxy, lower haloalkyl, lower haloalkoxy or lower alkylsulfonyloxy; R2 is hydrogen atom, lower alkyl, lower alkenyl, lower alkoxyalkyl, or lower alkylcarboxyl; X is oxygen atom or sulfur atom; Y is oxygen atom or sulfur atom; R3 is lower alkenyl or lower alknyl having unsubtituted or substituted radical (the substituted radical is hydroxy, halogen, lower alkoxy or the like) or lower cycloalkenyl].


(1)

CLAIMS

[Claim(s)]
[Claim 1]General formula (I)



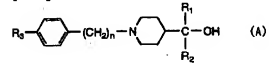
among [type, two R_1 may be the same or may differ — an each hydrogen atom. A halogen atom, a low-grade alkyl group, a lower alkoxy group, a low-grade halo alkyl group, A low-grade haloalkoxy group or a low-grade alkylsulfonyloxy group is shown, R_2 shows a hydrogen atom, a low-grade alkyl group, a low-grade alkenyl group, a low-grade alkoxyalkyl group, or a low-grade alkyl carbonyl group, X shows an oxygen atom or a sulfur atom, and Y shows an oxygen atom or a sulfur atom, A low-grade alkenyl group which R_3 is unsubstituted or has a substituent, or a low-grade alkynyl group (the aforementioned substituent) A hydroxy group, a halogen atom, a lower alkoxy group, a low-grade haloalkoxy group, A low-grade alkylthio group and low-grade alkyl sulfinyl group, a low-grade alkyl sulfonyl group, A low-grade cycloalkyl group and low-grade alkoxy alkoxy group, an amino group, a lower alkylamino group, A piperidine derivative expressed with being a low-grade dialkylamino group, a low-grade alkoxycarbonyl group, a nitro group, a cyano group, a trimethylsilyl group, or a phenyl group, or a low-grade cycloalkenyl group being shown], its N-oxide object, or its salt.
[Claim 2]two R_1 being the same, or differing, and in general formula (I), A halogen atom, a low-grade halo alkyl group, or a low-grade haloalkoxy group is shown respectively, R_2 shows a hydrogen atom, X shows an oxygen atom, and Y shows an oxygen atom, A low-grade alkenyl group which R_3 is unsubstituted or has a substituent, or a low-grade alkynyl group (the aforementioned substituent) a halogen atom, lower alkoxy group, low-grade haloalkoxy group, low-grade alkylthio group, and low-grade alkoxy alkoxy group, a low-grade dialkylamino group, or a low-grade alkoxycarbonyl group — it is — the shown piperidine derivative according to claim 1, its N-oxide object, or its salt.
[Claim 3]two R_1 being the same, or differing, and in general formula (I), A halogen atom, a low-grade halo alkyl group, or a low-grade haloalkoxy group is shown respectively, The piperidine derivative according to claim 1 which R_2 shows a hydrogen atom, X shows an oxygen atom, and Y shows an oxygen atom, and is a low-grade alkenyl group or a low-grade alkynyl group unsubstituted in R_3, its N-oxide object, or its salt.
[Claim 4]The piperidine derivative according to claim 1 which is N-[4-(2-propenyloxy

carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, its N-oxide object, or its salt.
[Claim 5]The piperidine derivative according to claim 1 which is N-[4-(2-propenyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methylphenyl)hydroxymethyl] piperidine, its N-oxide object, or its salt.
[Claim 6]The piperidine derivative according to claim 1 which is N-[4-(1-methyl-2-propenyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, its N-oxide object, or its salt.
[Claim 7]The piperidine derivative according to claim 1 which is N-[4-(2-propynyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, its N-oxide object, or its salt.
[Claim 8]The piperidine derivative according to claim 1 which is N-[4-(2-butynyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, its N-oxide object, or its salt.
[Claim 9]The piperidine derivative according to claim 1 which is N-[4-(1-methyl-2-propynyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, its N-oxide object, or its salt.
[Claim 10]The piperidine derivative according to claim 1 which is N-[4-(3-butynyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, its N-oxide object, or its salt.
[Claim 11]The piperidine derivative according to claim 1 which is N-[4-(N'-methyl-N'-(2-propenyloxy carbonyl) amino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, its N-oxide object, or its salt.
[Claim 12]An insecticide, miticide, or a nematicide which contains the piperidine derivative according to any one of claims 1 to 11, its N-oxide object, or its salt as an active principle.

[Translation done.]

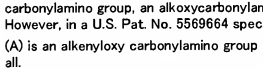
DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to the insecticide containing a new piperidine derivative useful as an insecticide, and this derivative as an active ingredient.
[0002]
[Description of the Prior Art]As a compound similar to the piperidine derivative of general formula (I) by this invention, the following are publicly known.
[0003](1) The compound shown in a U.S. Pat. No. 5569664 specification by the following general formula (A) is indicated to have insect-killing activity.
[0004]



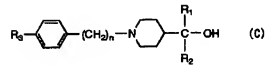
(A)

(n shows the integer of 1-3 among a formula, and R_1 and R_2 the phenyl group which may be replaced) [show and] R_3 shows a hydrogen atom, a hydroxy group, a halogen atom, an alkyl group, a halo alkyl group, an alkenyl group, an alkynyl group, an alkoxy group, an alkyl carbonylamino group, an alkoxycarbonylamino group, etc.
However, in a U.S. Pat. No. 5569664 specification, the compound whose R_3 in a general formula (A) is an alkenyloxy carbonylamino group and an alkynyloxy carbonylamino group is not written at all.
[0005](2) The compound shown in a U.S. Pat. No. 5639763 specification by the following general formula (B) is indicated to have insect-killing activity.
[0006]



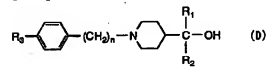
(B)

(n shows the integer of 1-3 among a formula, R_1 and R_2 show the phenyl group which may be replaced, and W and X become together and show $-OCH_2CH_2O-$, $-CH_2C(CH_3)_2O-$, etc.)
However, in a U.S. Pat. No. 5639763 specification, the compound whose X in a general formula (B) is an alkenyloxy carbonylamino group and an alkynyloxy carbonylamino group is not written at all.
[0007](3) The compound shown in the Patent Publication Heisei No. 505080 [nine to] gazette by the following general formula (C) is indicated to have insect-killing activity.
[0008]



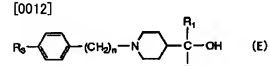
(C)

(n shows the integer of 1-3 among a formula, and R_1 and R_2 the phenyl group replaced by the halo alkyl group or the haloalkoxy group) [show and] R_3 shows a hydrogen atom, a hydroxy group, a halogen atom, an alkyl group, a halo alkyl group, an alkenyl group, an alkynyl group, an alkoxy group, an alkyl carbonylamino group, an alkoxycarbonylamino group, etc.
However, in the Patent Publication Heisei No. 505080 [nine to] gazette, the compound whose R_3 in a general formula (C) is an alkenyloxy carbonylamino group and an alkynyloxy carbonylamino group is not indicated at all.
[0009](4) The compound shown in a U.S. Pat. No. 6017931 specification by the following general formula (D) is indicated to have insect-killing activity.
[0010]



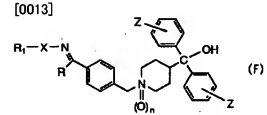
(D)

(n shows the integer of 1-3 among a formula, and R_1 and R_2 the phenyl group which may be replaced) [show and] R_3 An alkoxy group, a haloalkoxy group, an alkoxyalkyl group, A cycloalkyl alkoxy group, an alkoxycarbonylamino group, a haloalkoxy carbonylamino group, a cycloalkyl alkoxycarbonylamino group, a halo cycloalkyl alkoxycarbonylamino group, a cyano alkoxycarbonylamino group, etc. are shown.
However, in a U.S. Pat. No. 6017931 specification, the compound whose R_3 in a general formula (D) is an alkenyloxy carbonylamino group and an alkynyloxy carbonylamino group is not written at all.
[0011](5) The compound shown in the Patent Publication Heisei No. 509524 [11 to] gazette by the following general formula (E) is indicated to have insect-killing activity.
[0012]



(E)

(n shows the integer of 1-3 among a formula, R_1 and R_2 show the phenyl group or pyridyl group replaced by the halo alkyl group or the haloalkoxy group, and R_3 shows the heterocycle etc. of 5 members which may be replaced arbitrarily, or 6 members.)
(6) The compound shown in the International Patent Publication WO 99/No. 14193 gazette by the following general formula (F) is indicated to have insect-killing activity.
[0013]

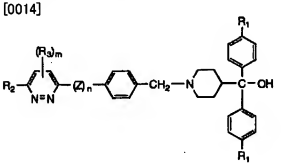


(F)

(R shows a hydrogen atom, a halogen atom, an alkyl group, etc. among a formula, R_1 shows a hydrogen atom, an alkyl group, a halo alkyl group, etc., X shows an oxygen atom and NR_2, n shows 0 or 1, and Z shows a halogen atom, a halo alkyl group, a haloalkoxy group, etc.)

(7) In JP,12-178272,A, the compound shown by the following general formula (G) is indicated to have insect-killing activity.

[0014]

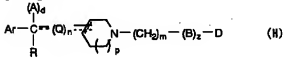


(G)

(R_1 among a formula a halogen atom, a C1-4 halo alkyl group, a C1-4 haloalkoxy group, etc.) [show and] R_2 A hydrogen atom, a hydroxy group, a halogen atom, C1-4 alkyl group, C1-4 alkoxy group etc. are shown, R_3 shows a halogen atom, C1-4 alkyl group, C1-4 alkoxy group, etc., Z shows oxygen atom and basis-S(O) p- (p is 0-2) etc., m shows the integer of 0-2, and n shows 0 or 1.

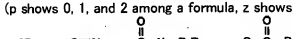
(8) In JP,62-169763,A, the compound shown by the following general formula (H) is indicated to have acrinia active works, such as activity, such as cardiovascular extension, antihistaminic activity, and gastric juice.

[0015]



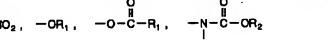
(H)

(p shows 0, 1, and 2 among a formula, z shows 0 or 1, and A is a hydrogen atom.)

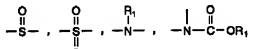


An example and m show 0-6, Q shows =CH-, $-CH_2-$, and -CH(OH)-, d and n show 0 or 1, and they are Ar, D, and R.

Pyridine, a thiophene, etc. are shown and X, Y, and Z are a hydrogen atom, a low-grade alkyl



group, and a halogen atom,

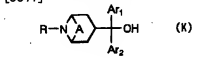


** is shown and B is an oxygen atom and a sulfur atom,

An example and R_1 show a hydrogen atom, a low-grade alkyl group, a phenyl group, and a phenyl low-grade alkyl group, and R_2 shows a low-grade alkyl group, a phenyl group, and a phenyl low-grade alkyl group. Carrying out a deer. There is no statement about insect-killing activity in JP,62-169763,A, and the compound whose above R_2 is a low-grade alkenyl group and a low-grade alkynyl group is not indicated at all.

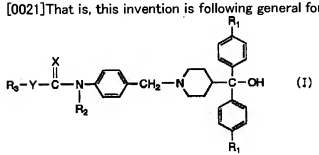
[0016](9) The compound shown in the British patent public presentation No. 2319524 gazette by

the following general formula (K) is indicated to have insect-killing activity.
[0017]



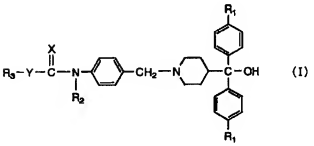
(K)

(A shows $-CH_2CH_2-$ etc. among a formula, Ar_1 and Ar_2 show the phenyl group etc. which may be replaced and R shows the aralkyl group etc. which may be replaced.)
[0018]
[Problem(s) to be Solved by the Invention]an object of this invention is to provide the piperidine derivative which has the insect-killing activity which was markedly alike and was excellent as compared with the conventional piperidine compound.
[0019]An object of this invention is for the extermination effect outstanding to the resistance noxious insect to be shown, and not to do damage to useful insects, to be safe for environment etc., and not to have an adverse effect, and to provide the piperidine derivative which is low toxicity also to mammalian.
[0020]
[Means for Solving the Problem]This invention persons compounded many new piperidine derivatives, in order to solve the above-mentioned technical problem, and they examined those insect-killing activity. As a result, a new piperidine derivative shown by following general formula (I) found out having the insect-killing activity excellent in a low dose. it found out having the immediate effect insect-killing activity which was markedly alike and was excellent in a low dose as compared with the conventional piperidine compound. This invention came to be completed based on these knowledge.
[0021]That is, this invention is following general formula (I).



(I)

among [type, two R_1 may be the same or may differ — an each hydrogen atom. A halogen atom, a low-grade alkyl group, a lower alkoxy group, a low-grade halo alkyl group, A low-grade haloalkoxy group or a low-grade alkylsulfonyloxy group is shown, R_2 shows a hydrogen atom, a low-grade alkyl group, a low-grade alkenyl group, a low-grade alkoxyalkyl group, or a low-grade alkyl carbonyl group, X shows an oxygen atom or a sulfur atom, and Y shows an oxygen atom or a sulfur atom, A low-grade alkenyl group which R_3 is unsubstituted or has a substituent, or a low-grade alkynyl group (the aforementioned substituent) A hydroxy group, a halogen atom, a lower alkoxy group, a low-grade haloalkoxy group, A low-grade alkylthio group and low-grade alkyl sulfinyl group, a low-grade alkyl sulfonyl group, A low-grade cycloalkyl group and low-grade alkoxy alkoxy group, an amino group, a lower alkylamino group, A piperidine derivative expressed with being a low-grade dialkylamino group, a low-grade alkoxycarbonyl group, a nitro group, a cyano group, a trimethylsilyl group, or a phenyl group, or a low-grade cycloalkenyl group being shown], its N-oxide object, or its salt is provided.
[0022]This invention is following general formula (I).



among [type, two R_1 may be the same or may differ — an each hydrogen atom. A halogen atom, a low-grade alkyl group, a lower alkoxy group, a low-grade halo alkyl group, A low-grade haloalkoxy group or a low-grade alkylsulfonyloxy group is shown, R_2 shows a hydrogen atom, a low-grade alkyl group, a low-grade alkenyl group, a low-grade alkoxyalkyl group, or a low-grade alkyl carbonyl group, X shows an oxygen atom or a sulfur atom, and Y carries out an oxygen atom or a sulfur atom, A low-grade alkenyl group which R_3 is unsubstituted or has a substituent, or a low-grade alkynyl group (the aforementioned substituent) A hydroxy group, a halogen atom, a lower alkoxy group, a low-grade haloalkoxy group, A low-grade alkylthio group and low-grade alkyl sulfinyl group, a low-grade alkyl sulfonyl group, A low-grade cycloalkyl group and low-grade alkoxy alkoxy group, an amino group, a lower alkylamino group, A low-grade dialkylamino group, a low-grade alkoxycarbonyl group, a nitro group, An insecticide, miticide, or a nematicide which contains a piperidine derivative expressed with being a cyano group, a trimethylsilyl group, or a phenyl group, or a low-grade cycloalkenyl group being shown], its N-oxide object, or its salt as an active principle is provided.

[0023]In above general formula (I), each basis shown by R_1, R_2, and R_3 can specifically mention the following.

[0024]As a "low-grade alkyl group", for example A methyl group, an ethyl group, n-propyl group, An isopropyl group, n-butyl group, an isobutyl group, a sec-butyl group, A tert-butyl group, n-pentyl group, an isopentyl group, 2-methylbutyl group, A neopentyl group, n-hexyl group, 4-methylpentyl group, 3-methylpentyl group, 2-methylpentyl group, a 3,3-dimethylbutyl group, an 1,1-dimethylbutyl group, A 1,3-dimethylbutyl group, a 2,3-dimethylbutyl group, 1-ethylbutyl group, A 1-methyl-1-ethylpropyl group, a 1,2-dimethylbutyl group, a 2-methyl-1-ethylpropyl group, Straight chain shape of 1-6 carbon numbers or branched state alkyl groups, such as a 2,2-dimethylbutyl group, can be mentioned, and they are a methyl group, an ethyl group, n-propyl group, an isopropyl group, n-butyl group, an isobutyl group, a sec-butyl group, and a tert-butyl group preferably.

[0025]As a "low-grade alkenyl group", for example A vinyl group, 1-propenyl group, 2-propenyl group, a 1-methyl-2-propenyl group, a 2-methyl-2-propenyl group, A 2-ethyl-2-propenyl group, 2-butenyl group, a 1-methyl-2-butenyl group, A 2-methyl-2-butenyl group, a 1-ethyl-2-butenyl group, 3-butenyl group, 2-pentenyl group, 3-pentenyl group, 4-pentenyl group, 2-hexenyl group, Can mention straight chain shape of 2-6 carbon numbers of 3-hexenyl group or 4-hexenyl group, or a branched state alkenyl group, and preferably, They are 1-propenyl group, 2-propenyl group, a 1-methyl-2-propenyl group, a 2-methyl-2-propenyl group, a 2-ethyl-2-propenyl group, 2-butenyl group, and 3-butenyl group.

[0026]As a "low-grade alkynyl group", for example 2-propynyl group, 1-methyl-2-propynyl group, 2-butynyl group, a 1-methyl-2-butynyl group, a 1-ethyl-2-butynyl group, 3-butynyl group, a 2-methyl-3-butynyl group, 2-pentynyl group, Straight chain shape of 3-6 carbon numbers or branched state alkynyl groups, such as 4-pentynyl group, 2-hexynil group, 3-hexynil group, 4-hexynil group, or 5-hexynil group, can be mentioned, Preferably, they are 2-propynyl group, 1-methyl-2-propynyl group, 2-butynyl group, and 3-butynyl group.

[0027]As a "low-grade cycloalkenyl group", for example 2-cyclopropenyl group, A cycloalkenyl group of 3-6 carbon numbers, such as 1-cyclo butenyl group, 2-cyclo butenyl group, 1-cyclopentenyl group, 2-cyclopentenyl group, 1-cyclohexenyl group, and 2-cyclohexenyl group,

can be mentioned.
[0028]As a "low-grade cycloalkyl group", for example A cyclopropyl group, a cyclobutyl group, A cyclopentylic group, a cyclohexyl group, 2-methylcyclopropyl group, A cycloalkyl group of 3-7 carbon numbers which may have the branched chain of 1-6 carbon numbers of 2-methyl cyclopentylic group or 2-methylcyclohexyl group can be mentioned, and it is a cyclopropyl group preferably.
[0029]As a "halogen atom", each atom of fluoride, chlorine, bromine, or iodine can be mentioned.

[0030]As a "low-grade halo alkyl group", for example A trifluoromethyl group, A fluoromethyl group, a chloromethyl group, a bromomethyl group, an iodomethyl group, Difluoromethyl group, a dichloromethyl group, a trichloromethyl group, 2-fluoroethyl group, 2-chloroethyl group, 2-bromoethyl group, 1-fluoroethyl group, An 1,1-difluoroethyl group, 2 and 2, 2-trifluoroethyl group, A pentafluoroethyl group, 1-fluoropropyl group, 3-fluoropropyl group, 2-chloropropyl group, 3-chloropropyl group, 3-iodopropyl group, A low-grade alkyl group which a halogen atom like fluoride, chlorine, bromine, or iodine combined with the aforementioned low-grade alkyl group of 1-fluorobutyl group, 4-fluorobutyl group, 1-chlorobutyl group, etc. can be mentioned, and it is a trifluoromethyl group preferably.
[0031]As a "lower alkoxy group", for example A methoxy group, an ethoxy basis, n-propoxy group, an isopropoxy group, n-butoxy group, an isobutoxy group, Straight chain shape of 1-6 carbon numbers of a sec-butoxy group, a tert-butoxy group, an n-pentyloxy group, an isopentyloxy group, an n-hexyloxy group, etc. or a branched state alkoxy group can be mentioned, and they are a methoxy group and an ethoxy basis preferably.
[0032]As a "low-grade haloalkoxy group", for example A fluoro methoxy group, A chloro methoxy group, a bromo methoxy group, an iodo methoxy group, a difluoro methoxy group, A trifluoro methoxy group, 1-fluoroethoxy group, 2-fluoroethoxy group, 2-chloroethoxy group, a 2,2,2-trifluoroethoxy group, a pentafluoro ethoxy basis, 1-fluoro propoxy group, 3-fluoro propoxy group, 2-chloro propoxy group, Straight chain shape of 1-6 carbon numbers or a branched state haloalkoxy group of 3-chloro propoxy group, 1-fluorobutoxy group, 4-fluorobutoxy group, 1-chlorobutoxy group, etc. can be mentioned, and they are a trifluoro methoxy group and a difluoro methoxy group preferably.
[0033]As a "low-grade alkylthio group", for example A methylthio group, an ethyl thio group, Straight chain shape of 1-4 carbon numbers or branched state alkylthio groups, such as n-propyl thio group, an isopropyl thio group, n-butyl thio group, an isobutyl thio group, a sec-butyl thio group, and a tert-butyl thio group, can be mentioned, and it is a methylthio group preferably.
[0034]As a "low-grade alkyl sulfinyl group", for example A methyl sulfinyl group, An ethyl sulfinyl group, n-propyl sulfinyl group, an isopropyl sulfinyl group, n-butyl sulfinyl group, an isobutyl sulfinyl group, a sec-butyl sulfinyl group, Alkyl parts, such as a tert-butyl sulfinyl group, can mention straight chain shape of 1-4 carbon numbers, or a branched state alkyl sulfinyl group, and are methyl sulfinyl groups preferably.
[0035]As a "low-grade alkyl sulfonyl group", for example A methylsulfonyl group, An ethyl sulfonyl group, n-propyl sulfonyl group, an isopropylsulfonyl group, Alkyl parts, such as n-butyl sulfonyl group, an isobutyl sulfonyl group, a sec-butyl sulfonyl group, and a tert-butyl sulfonyl group, can mention straight chain shape of 1-4 carbon numbers, or a branched state alkyl sulfonyl group, and are methylsulfonyl groups preferably.
[0036]As a "low-grade alkoxy alkoxy group", for example A methoxy methoxy group, An ethoxy methoxy group, n-propoxy methoxy group, an isopropoxy methoxy group, n-butoxy methoxy group, an isobutoxy methoxy group, a sec-butoxy methoxy group, A tert-butoxy methoxy group, an n-pentyloxy methoxy group, an isopentyloxy methoxy group, An n-hexyloxy methoxy group, a methoxyethoxy group, an ethoxyethoxy basis, n-propoxyethoxy group, an isopropoxy ethoxy basis, n-butoxyethoxy group, An isobutoxyethoxy basis, a sec-butoxyethoxy group, a tert-butoxyethoxy group, A methoxy propoxy group and ethoxy propoxy group, n-propoxy propoxy group, Each alkoxy portions, such as an isopropoxy propoxy group and n-butoxy propoxy group, can mention straight chain shape of 1-6 carbon numbers, or a branched state alkoxy alkoxy group, and are methoxy methoxy group and ethoxy methoxy groups preferably.

[0037]As a "lower alkylamino group", for example A methylamino group, an ethylamino group, n-propylamino group, an isopropylamino group, n-butylamino group, An isobutylamino group, a sec-butylamino group, a tert-butylamino group, n-pentylamino group, an isopentylamino group, 2-methylbutylamino group, A neopentyl amino group, n-hexylamino group, 4-methyl pentylamino group, 3-methyl pentylamino group, 2-methyl pentylamino group, a 3,3-dimethyl butylamino group, An 1,1-dimethyl butylamino group, a 1,3-dimethyl butylamino group, A 2,3-dimethyl butylamino group, 1-ethyl butylamino group, a 1-methyl-1-ethyl propylamino group, Straight chain shape of 1-6 carbon numbers of a 1,2-dimethyl butylamino group, a 2-methyl-1-ethyl propylamino group, a 2,2-dimethyl butylamino group, etc. or a branched state alkylamino group can be mentioned, and it is a methylamino group preferably.
[0038]As a "low-grade dialkylamino group", for example A dimethylamino group, A diethylamino group, a JI n-propylamino group, a diisopropylamino group, A di-n-butyl amino group, an ethyl methylamino group, n-propyl methylamino group, An isopropyl methylamino group, n-butyl methylamino group, an ethyl n-propylamino group, Each alkyl parts, such as an ethyl isopropylamino group and an ethyl n-butylamino group, can mention straight chain shape of 1-6 carbon numbers, or a branched state dialkylamino group, and are dimethylamino groups preferably.
[0039]As a "low-grade alkoxycarbonyl group", for example A methoxycarbonyl group, An ethoxycarbonyl group, n-carbopropoxy group, an isopropoxycarbonyl group, n-butoxycarbonyl group, an isocarbobutoxy group, a sec-butoxycarbonyl group, Alkoxy portions, such as a tert-butoxycarbonyl group, can mention straight chain shape of 1-6 carbon numbers, or a branched state alkoxycarbonyl group, and are a methoxycarbonyl group and an ethoxycarbonyl group preferably.
[0040]As a "low-grade alkoxyalkyl group", for example A methoxymethyl group, An ethoxymethyl group, n-propoxymethyl group, an isopropoxy methyl group, n-butoxymethyl group, an isobutoxymethyl group, a sec-butoxymethyl group, A tert-butoxymethyl group, n-pentyl oxymethyl group, an isopentyl oxymethyl group, An n-hexyl oxymethyl group, methoxy ethyl group, and ethoxyethyl group, n-propoxyethyl group, an isopropoxy ethyl group, n-butoxyethyl group, An isobutoxyethyl group, a sec-butoxyethyl group, a tert-butoxyethyl group, A methoxy propyl group and ethoxypropyl group, n-propoxypropyl group, Alkoxy portions, such as an isopropoxy propyl group and n-butoxypropyl group, can mention straight chain shape of 1-6 carbon numbers, or a branched state alkoxyalkyl group, and are a methoxymethyl group and an ethoxymethyl group preferably.
[0041]As a "low-grade alkyl carbonyl group", for example A methyl carbonyl group, An ethyl carbonyl group, n-propylcarbonyl group, an isopropyl carbonyl group, Alkyl parts, such as n-butyl carbonyl group, an isobutyl carbonyl group, a sec-butyl carbonyl group, and a tert-butyl carbonyl group, can mention straight chain shape of 1-6 carbon numbers, or a branched state alkyl carbonyl group, and are methyl carbonyl groups preferably.
[0042]Next, a compound shown in the following table 1 as an example of a compound contained in general formula (I) by this invention can be mentioned. However, this invention is not limited to these examples.
[0043]

【表1】

表1

化合物番号	R_1	R_2	R_3	X	Y	形態
1	CF_2O	H	$CH_2=CHCH_2-$	O	O	
2	CF_2O	H	$CH_2=CHCH_2-$	O	O	N－オキシド
3	CF_2O	H	$CH_2=CHCH_2-$	O	O	塩酸塩
4	CF_2O	H	$CH_2=CHCH_2-$	O	O	D－グルクロン酸塩
5	CF_2O	H	$CH_2=CHCH_2-$	O	O	エタンスルホン酸塩
6	CF_2O	H	$CH_2=CHCH_2-$	O	O	N－オキシド塩酸塩
7	CF_2O	H	$CH_2=CHCH_2-$	O	O	N－オキシドエタンスルホン酸塩
8	CHF_2O	H	$CH_2=CHCH_2-$	O	O	
9	CHF_2O	H	$CH_2=CHCH_2-$	O	O	N－オキシド
10	CHF_2O	H	$CH_2=CHCH_2-$	O	O	塩酸塩
11	CHF_2O	H	$CH_2=CHCH_2-$	O	O	エタンスルホン酸塩
12	CF_3	H	$CH_2=CHCH_2-$	O	O	
13	CF_3	H	$CH_2=CHCH_2-$	O	O	N－オキシド
14	CF_3	H	$CH_2=CHCH_2-$	O	O	塩酸塩
15	CF_3	H	$CH_2=CHCH_2-$	O	O	サリチル酸塩
16	CF_3	H	$CH_2=CHCH_2-$	O	O	2－ヒドロキシエタンスルホン酸塩
17	CF_2O, CF_3	H	$CH_2=CHCH_2-$	O	O	
18	CF_2O, CHF_2O	H	$CH_2=CHCH_2-$	O	O	
19	CF_2O, Cl	H	$CH_2=CHCH_2-$	O	O	
20	CF_2O, F	H	$CH_2=CHCH_2-$	O	O	
21	F	H	$CH_2=CHCH_2-$	O	O	
22	Cl	H	$CH_2=CHCH_2-$	O	O	
23	Br	H	$CH_2=CHCH_2-$	O	O	
24	I	H	$CH_2=CHCH_2-$	O	O	
25	H	H	$CH_2=CHCH_2-$	O	O	
26	CH_3	H	$CH_2=CHCH_2-$	O	O	
27	tBu	H	$CH_2=CHCH_2-$	O	O	

【表2】

表1(続き1)

化合物番号	R_1	R_2	R_3	X	Y	形態
28	CH_3O	H	CH_2=$CHCH_2$−	O	O	
29	CH_3SO_2O	H	CH_2=$CHCH_2$−	O	O	
30	CF_3O	CH_3	CH_2=$CHCH_2$−	O	O	
31	CF_3O	CH_3	CH_2=$CHCH_2$−	O	O	N−オキシド
32	CF_3O	CH_3	CH_2=$CHCH_2$−	O	O	エタンスルホン酸塩
33	CHF_2O	CH_3	CH_2=$CHCH_2$−	O	O	
34	CF_3	CH_3	CH_2=$CHCH_2$−	O	O	
35	CF_3O	CH_3OCH_2	CH_2=$CHCH_2$−	O	O	
36	CF_3O	$EtOCH_2$	CH_2=$CHCH_2$−	O	O	
37	CF_3O	CH_2=$CHCH_2$−	CH_2=$CHCH_2$−	O	O	
38	CF_3O	CH_3−C(=O)−	CH_2=$CHCH_2$−	O	O	
39	CF_3O	H	CH_2=$CHCH_2$−	S	O	
40	CF_3O	H	CH_2=$CHCH_2$−	O	S	
41	CF_3O	H	FCH=$CHCH_2$−	O	O	
42	CF_3O	H	ClCH=$CHCH_2$−	O	O	
43	CF_3O	H	ICH=$CHCH_2$−	O	O	
44	CF_3O	H	CH_2=C(Cl)CH_2−	O	O	
45	CF_3O	H	CH_2=C(Br)CH_2−	O	O	
46	CF_3O	H	CH_2=C(I)CH_2−	O	O	
47	CF_3O	H	CH_2=C(F)CH−	O	O	
48	CF_3O	H	Cl−CH=C(Cl)CH_2−	O	O	
49	CF_3O	H	Br−CH=C(Br)CH_2−	O	O	

【表3】

表1(続き2)

化合物番号	R_1	R_2	R_3	X	Y	形態
50	CF_3O	H		O	O	
51	CF_3O	H		O	O	N−オキシド
52	CF_3O	H		O	O	エタンスルホン酸塩
53	CF_3O	H		O	O	
54	CF_3O	H		O	O	
55	CF_3O	H		O	O	
56	CF_3O	H		O	O	
57	CF_3O	H	$CH_3O-CH=CHCH_2-$	O	O	
58	CF_3O	H	$EtO-CH=CHCH_2-$	O	O	
59	CF_3O	H		O	O	
60	CF_3O	H		O	O	N−オキシド
61	CF_3O	H		O	O	
62	CF_3O	H		O	O	
63	CF_3O	H		O	O	
64	CF_3O	H	$CH_3S-CH=CHCH_2-$	O	O	
65	CF_3O	H	$EtS-CH=CHCH_2-$	O	O	

【表4】

表1(続き3)

化合物番号	R_1	R_2	R_3	X	Y	形態
66	CF_3O	H	CH_3-S $CH_2=CCH_2-$	O	O	
67	CF_3O	H	$Et-S$ $CH_2=CCH_2-$	O	O	
68	CF_3O	H	$EtSO-CH=CHCH_2-$	O	O	
69	CF_3O	H	$CH_3SO_2-CH=CHCH_2-$	O	O	
70	CF_3O	H	$CH_3-Si(CH_3)_2-CH=CHCH_2-$	O	O	
71	CF_3O	H	NO_2 $CH_2=CCH_2-$	O	O	
72	CF_3O	H	$NC-CH=CHCH_2-$	O	O	
73	CF_3O	H		O	O	
74	CF_3O	H	$CH_3O_2C-CH=CHCH_2-$	O	O	
75	CF_3O	H	CH_3O_2C $CH_2=CHCH-$	O	O	
76	CF_3O	H	$NH_2-CH=CHCH_2-$	O	O	
77	CF_3O	H	$CH_3NH-CH=CHCH_2-$	O	O	
78	CF_3O	H		O	O	
79	CF_3O	H		O	O	塩酸塩
80	CF_3O	H	$HO-CH=CHCH_2-$	O	O	
81	CF_3O	H	$CF_3O-CH=CHCH_2-$	O	O	
82	CF_3O	H		O	O	

【表5】

表1(続き4)

化合物番号	R_1	R_2	R_3	X	Y	形態
83	CF_3O	H	$CH_2=CHCH-$	O	O	
84	CF_3O	H	$CH_2=CHCH-$	O	O	Nーオキシド
85	CF_3O	H	$CH_2=CHCH-$	O	O	塩酸塩
86	CHF_2O	H	$CH_2=CHCH-$	O	O	
87	CF_3	H	$CH_2=CHCH-$	O	O	
88	CF_3O, CF_3	H	$CH_2=CHCH-$	O	O	
89	CF_3	H	$CH_2=CHCH-$	O	S	
90	CF_3	CH_3	$CH_2=CHCH-$	O	O	
91	F	H	$CH_2=CHCH-$	O	O	
92	Cl	H	$CH_2=CHCH-$	O	O	
93	Br	H	$CH_2=CHCH-$	O	O	
94	iPr	H	$CH_2=CHCH-$	O	O	
95	CH_3SO_2O	H	$CH_2=CHCH-$	O	O	
96	CF_3O	H	$CH_2=CHCH-$	O	O	
97	CF_3O	H	$CH_2=CHCH-$	O	O	

【表6】

表1(続き5)

化合物番号	R_1	R_2	R_3	X	Y	形態
98	CF_3O	H	$CH_2=CHCH(CH_2OEt)-$	O	O	
99	CF_3O	H	$CH_2=CHCH(CH_2SCH_3)-$	O	O	
100	CF_3O	H	$F-CH=CHCH(CH_3)-$	O	O	
101	CF_3O	H	$Cl-CH=CHCH(CH_3)-$	O	O	
102	CF_3O	H	$Cl_2C=CHCH(CH_3)-$	O	O	
103	CF_3O	H	$CH_3O-CH=CHCH(CH_3)-$	O	O	
104	CF_3O	H	$CH_2=C(CH_3)CH_2-$	O	O	
105	CF_3O	H	$F-CH=C(CH_3)CH_2-$	O	O	
106	CF_3O	H	$Cl-CH=C(CH_3)CH_2-$	O	O	
107	CF_3O	H	$Br-CH=C(CH_3)CH_2-$	O	O	
108	CF_3O	H	$I-CH=C(CH_3)CH_2-$	O	O	
109	CF_3O	H	$Cl_2C=C(CH_3)CH_2-$	O	O	
110	CF_3O	H	$Br_2C=C(CH_3)CH_2-$	O	O	

【表7】

表1(続き6)

化合物番号	R_1	R_2	R_3	X	Y	形態
111	CF_3O	H	$CH_3O-CH=\overset{CH_3}{\overset{\vert}{C}}CH_2-$	O	O	
112	CF_3O	H	$CH_3O-CH=\overset{CH_3}{\overset{\vert}{C}}CH_2-$	O	O	N-オキシド
113	CF_3O	H	$CH_3O-CH=\overset{CH_3}{\overset{\vert}{C}}CH_2-$	O	O	エタンスルホン酸塩
114	CF_3O	H	$CH_3S-CH=\overset{CH_3}{\overset{\vert}{C}}CH_2-$	O	O	
115	CF_3O	H	$CH_3O-\overset{CH_2}{\overset{\vert}{C}}H_2=CCH_2-$	O	O	
116	CF_3O	H	$EtO-\overset{CH_2}{\overset{\vert}{C}}H_2=CCH_2-$	O	O	
117	CF_3O	H	$NC-CH=\overset{CH_3}{\overset{\vert}{C}}CH_2-$	O	O	
118	CF_3O	H	$\begin{matrix}CH_3\\ \diagdown\\ CH_3\end{matrix}N-CH=\overset{CH_3}{\overset{\vert}{C}}CH_2-$	O	O	
119	CF_3O	H	$CH_3CH=CHCH_2-$	O	O	
120	CF_3O	H	$F-CH_2CH=CHCH_2-$	O	O	
121	CF_3O	H	$Cl-CH_2CH=CHCH_2-$	O	O	
122	CF_3O	H	$Br-CH_2CH=CHCH_2-$	O	O	
123	CF_3O	H	$I-CH_2CH=CHCH_2-$	O	O	
124	CF_3O	H	$CH_3\overset{Cl}{\overset{\vert}{C}}=CHCH_2-$	O	O	
125	CF_3O	H	$CH_3CH=\overset{Br}{\overset{\vert}{C}}CH_2-$	O	O	
126	CF_3O	H	$CH_3-\overset{F}{\overset{\vert}{C}}=\overset{F}{\overset{\vert}{C}}CH_2-$	O	O	
127	CF_3O	H	$CF_3CH=CHCH_2-$	O	O	

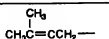
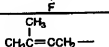
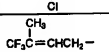
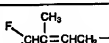
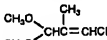
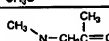
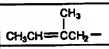
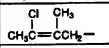
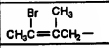
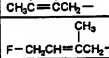
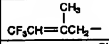
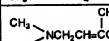
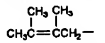
【表8】

表1(続き7)

化合物番号	R_1	R_2	R_3	X	Y	形態
128	CF_3O	H	$HO-CH_2CH=CHCH_2-$	O	O	
129	CF_3O	H	$CH_3O-CH_2CH=CHCH_2-$	O	O	
130	CF_3O	H	$EtO-CH_2CH=CHCH_2-$	O	O	
131	CF_3O	H	$ClCH_2O-CH_2CH=CHCH_2-$	O	O	
132	CF_3O	H	$CH_3C(=CHCH_2-)-O-CH_3$	O	O	
133	CF_3O	H	$CH_3C(=CHCH_2-)-O-Et$	O	O	
134	CF_3O	H	$CH_3CH=CCH_2-O-CH_3$	O	O	
135	CF_3O	H	$CH_3OCH_2O-CH_2CH=CHCH_2-$	O	O	
136	CF_3O	H	$NH_2-CH_2CH=CHCH_2-$	O	O	
137	CF_3O	H	$(CH_3)_2N-CH_2CH=CHCH_2-$	O	O	
138	CF_3O	H	$(Et)_2N-CH_2CH=CHCH_2-$	O	O	
139	CF_3O	H	$NO_2-CH_2CH=CHCH_2-$	O	O	
140	CF_3O	H	$CH_3C(=CHCH_2-)-CH_3$	O	O	
141	CF_3O	H	$CH_3C(=CHCH_2-)-CH_3$	O	O	N-オキシド
142	CF_3O	H	$F-CH_2C(=CHCH_2-)-CH_3$	O	O	
143	CF_3O	H	$Cl-CH_2C(=CHCH_2-)-CH_3$	O	O	
144	CF_3O	H	$Br-CH_2C(=CHCH_2-)-CH_3$	O	O	

【表9】

表1(続き8)

化合物番号	R_1	R_2	R_3	X	Y	形態
145	CF_3O	H		O	O	
146	CF_3O	H		O	O	
147	CF_3O	H		O	O	
148	CF_3O	H		O	O	
149	CF_3O	H		O	O	
150	CF_3O	H		O	O	
151	CF_3O	H		O	O	
152	CF_3O	H		O	O	
153	CF_3O	H		O	O	
154	CF_3O	H		O	O	
155	CF_3O	H		O	O	
156	CF_3O	H		O	O	
157	CF_3O	H		O	O	

【表 10】

表1(続き9)

化合物番号	R_1	R_2	R_3	X	Y	形態
158	CF_3O	H	$CH_3CH=\overset{Et}{\overset{\vert}{C}}CH_2-$	O	O	
159	CF_3O	H	$CH_3CH=\overset{BrCH_2-CH_2}{\overset{\vert}{C}}CH_2-$	O	O	
160	CF_3O	H	$CH_2=CH\overset{CH_3}{\overset{\vert}{C}}(\overset{CH_3}{\overset{\vert}{CH_3}})-$	O	O	
161	CF_3O	H	$CH_2=\overset{Et}{\overset{\vert}{C}}H-$	O	O	
162	CF_3O	H	$CH_2=\overset{nPr}{\overset{\vert}{C}}H-$	O	O	
163	CF_3O	H	$nPr-CH=CHCH_3-$	O	O	
164	CF_3O	H	$Et-CH=CHCH_2CH_2-$	O	O	
165	CF_3O	H	$CH_3-CH=CHCH_2CH_2CH_2-$	O	O	
166	CF_3O	H	$CH_2=CH(CH_2)_4-$	O	O	
167	CF_3O	H	$CH_2=\overset{CH_3}{\overset{\vert}{C}}CH_2CH_2-$	O	O	
168	CF_3O	H	$CH_3CH=CHCH=CHCH_2-$	O	O	
169	CF_3O	H		O	O	
170	CF_3O	H		O	O	
171	CF_3O	H		O	O	
172	CF_3O	H		O	O	
173	CF_3O	H		O	O	
174	CF_3O	H	$CH\equiv CCH_2-$	O	O	
175	CF_3O	H	$CH\equiv CCH_2-$	O	O	N-オキシド

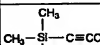
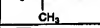
【表 11】

表1(続き 10)

化合物番号	R_1	R_2	R_3	X	Y	形態
176	CF_3O	H	$CH{\equiv}CCH_2-$	O	O	エタンスルホン酸塩
177	CHF_2O	H	$CH{\equiv}CCH_2-$	O	O	
178	CF_3	H	$CH{\equiv}CCH_2-$	O	O	
179	F	H	$CH{\equiv}CCH_2-$	O	O	
180	Cl	H	$CH{\equiv}CCH_2-$	O	O	
181	Br	H	$CH{\equiv}CCH_2-$	O	O	
182	I	H	$CH{\equiv}CCH_2-$	O	O	
183	H	H	$CH{\equiv}CCH_2-$	O	O	
184	CH_3	H	$CH{\equiv}CCH_2-$	O	O	
185	tBu	H	$CH{\equiv}CCH_2-$	O	O	
186	CH_3O	H	$CH{\equiv}CCH_2-$	O	O	
187	CH_3SO_2O	H	$CH{\equiv}CCH_2-$	O	O	
188	CF_3O, CF_3	H	$CH{\equiv}CCH_2-$	O	O	
189	CF_3O, Cl	H	$CH{\equiv}CCH_2-$	O	O	
190	CF_3O	CH_3	$CH{\equiv}CCH_2-$	O	O	
191	CF_3O	CH_2OCH_2	$CH{\equiv}CCH_2-$	O	O	
192	CF_3O	$CH_2{=}CHCH_2-$	$CH{\equiv}CCH_2-$	O	O	
193	CF_3O	$CH_3-\overset{O}{\overset{\|}{C}}-$	$CH{\equiv}CCH_2-$	O	O	
194	CF_3O	H	$CH{\equiv}CCH_2-$	S	O	
195	CF_3O	H	$CH{\equiv}CCH_2-$	O	S	
196	CF_3O	H	$F-C{\equiv}CCH_2-$	O	O	
197	CF_3O	H	$Cl-C{\equiv}CCH_2-$	O	O	

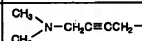
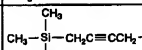
【表 12】

表1(続き 11)

化合物番号	R_1	R_2	R_3	X	Y	形態
198	CF_3O	H	$Br-C\equiv CCH_2-$	O	O	
199	CF_3O	H	$I-C\equiv CCH_2-$	O	O	
200	CF_3O	H	$CH_3O-C\equiv CCH_2-$	O	O	
201	CF_3O	H	$EtO-C\equiv CCH_2-$	O	O	
202	CF_3O	H		O	O	
203	CF_3O	H		O	O	
204	CF_3O	H	$CH_3C\equiv CCH_2-$	O	O	
205	CF_3O	H	$CH_3C\equiv CCH_2-$	O	O	N-オキシド
206	CHF_2O	H	$CH_3C\equiv CCH_2-$	O	O	
207	CHF_2O	H	$CH_3C\equiv CCH_2-$	O	O	エタンスルホン酸塩
208	CF_3	H	$CH_3C\equiv CCH_2-$	O	O	
209	CF_3	H	$CH_3C\equiv CCH_2-$	O	S	
210	CF_3	CH_3	$CH_3C\equiv CCH_2-$	O	O	
211	F	H	$CH_3C\equiv CCH_2-$	O	O	
212	Cl	H	$CH_3C\equiv CCH_2-$	O	O	
213	Br	H	$CH_3C\equiv CCH_2-$	O	O	
214	iPr	H	$CH_3C\equiv CCH_2-$	O	O	
215	CH_3SO_2O	H	$CH_3C\equiv CCH_2-$	O	O	
216	CF_3O	H	$F-CH_2C\equiv CCH_2-$	O	O	
217	CF_3O	H	$Cl-CH_2C\equiv CCH_2-$	O	O	
218	CF_3O	H	$Br-CH_2C\equiv CCH_2-$	O	O	

【表 13】

表1(続き 12)

化合物番号	R_1	R_2	R_3	X	Y	形態
219	CF_3O	H	$CF_3-C\equiv CCH_2-$	O	O	
220	CF_3O	H	$CH_3O-CH_2C\equiv CCH_2-$	O	O	
221	CF_3O	H	nPrO$-CH_2C\equiv CCH_2-$	O	O	
222	CF_3O	H	nPrO$-CH_2C\equiv CCH_2-$	O	O	塩酸塩
223	CF_3O	H	$CF_3O-CH_2C\equiv CCH_2-$	O	O	
224	CF_3O	H	$CH_3S-CH_2C\equiv CCH_2-$	O	O	
225	CF_3O	H	$NH_2-CH_2C\equiv CCH_2-$	O	O	
226	CF_3O	H	$CH_3NH-CH_2C\equiv CCH_2-$	O	O	
227	CF_3O	H		O	O	
228	CF_3O	H		O	O	
229	CF_3O	H	$CH\equiv CCH_2CH_2-$	O	O	
230	CF_3O	H	$CH\equiv CCH_2CH_2-$	O	O	N−オキシド
231	CF_3O	H	$CH\equiv CCH_2CH_2-$	O	O	塩酸塩
232	CHF_2O	H	$CH\equiv CCH_2CH_2-$	O	O	
233	CF_3	H	$CH\equiv CCH_2CH_2-$	O	O	
234	CF_3	H	$CH\equiv CCH_2CH_2-$	O	O	N−オキシド
235	F	H	$CH\equiv CCH_2CH_2-$	O	O	
236	Cl	H	$CH\equiv CCH_2CH_2-$	O	O	
237	Br	H	$CH\equiv CCH_2CH_2-$	O	O	
238	I	H	$CH\equiv CCH_2CH_2-$	O	O	
239	H	H	$CH\equiv CCH_2CH_2-$	O	O	

【表 14】

表1(続き 13)

化合物番号	R_1	R_2	R_3	X	Y	形態
240	CH_3	H	$CH{\equiv}CCH_2CH_2-$	O	O	
241	tBu	H	$CH{\equiv}CCH_2CH_2-$	O	O	
242	CH_3O	H	$CH{\equiv}CCH_2CH_2-$	O	O	
243	CH_3SO_2O	H	$CH{\equiv}CCH_2CH_2-$	O	O	
244	CF_3O	CH_3	$CH{\equiv}CCH_2CH_2-$	O	O	
245	CF_3O	CH_3OCH_2	$CH{\equiv}CCH_2CH_2-$	O	O	
246	CF_3O	$CH_2{=}CHCH_2-$	$CH{\equiv}CCH_2CH_2-$	O	O	
247	CF_3O	$CH_3-\overset{O}{\overset{\|}{C}}-$	$CH{\equiv}CCH_2CH_2-$	O	O	
248	CF_3O	H	$CH{\equiv}CCH_2CH_2-$	S	O	
249	CF_3O	H	$CH{\equiv}CCH_2CH_2-$	O	S	
250	CF_3O	H	$CH{\equiv}\overset{CH_3}{\overset{\|}{C}}H-$	O	O	
251	CF_3O	H	$CH{\equiv}\overset{CH_3}{\overset{\|}{C}}H-$	O	O	N−オキシド
252	CF_3O	CH_3	$CH{\equiv}\overset{CH_3}{\overset{\|}{C}}H-$	O	O	
253	CHF_2O	H	$CH{\equiv}\overset{CH_3}{\overset{\|}{C}}H-$	O	O	
254	CF_3	H	$CH{\equiv}\overset{CH_3}{\overset{\|}{C}}H-$	O	O	
255	CF_3	H	$CH{\equiv}\overset{CH_3}{\overset{\|}{C}}H-$	O	S	
256	CF_3	CH_3	$CH{\equiv}\overset{CH_3}{\overset{\|}{C}}H-$	O	O	

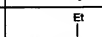
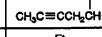
【表 15】

表1(続き 14)

化合物番号	R_1	R_2	R_3	X	Y	形態
257	F	H		O	O	
258	F	H		O	O	塩酸塩
259	Cl	H		O	O	
260	Cl	H		O	O	塩酸塩
261	Br	H		O	O	
262	IPr	H		O	O	
263	CH_3SO_2O	H		O	O	
264	CF_3O	H	$CH{\equiv}CCH_2CH_2CH_2-$	O	O	
265	CF_3O	H	$CH_3C{\equiv}CCH_2CH_2-$	O	O	
266	CF_3O	H	$EtC{\equiv}CCH_2-$	O	O	
267	CF_3O	H		O	O	
268	CF_3O	H		O	O	
269	CF_3O	H		O	O	
270	CF_3O	H	$CH{\equiv}C(CH_2)_4-$	O	O	

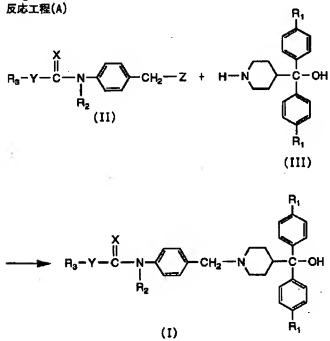
【表 16】

表1(続き 15)

化合物番号	R_1	R_2	R_3	X	Y	形態
271	CF_3O	H	$CH_3C{\equiv}CCH_2CH_2CH_2-$	O	O	
272	CF_3O	H	$EtC{\equiv}CCH_2CH_2-$	O	O	
273	CF_3O	H	$nPrC{\equiv}CCH_2-$	O	O	
274	CF_3O	H		O	O	
275	CF_3O	H		O	O	
276	CF_3O	H		O	O	
277	CF_3O	H		O	O	

[0044]
[Embodiment of the Invention]The manufacturing method of the piperidine derivative of general formula (I) by this invention is explained in detail.
[0045]The piperidine derivative of general formula (I) by this invention can be manufactured by the method of the following reaction process (A), for example.
[0046]

反応工程(A)

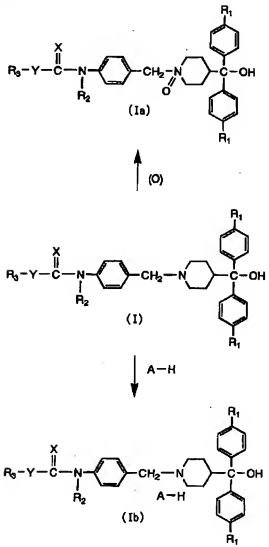


(II) (III) (I)

(R_1, R_2, R_3, X, and Y show the same meaning as the above among a formula, and Z shows a halogen atom.)

That is, in a reaction process (A), the piperidine derivative shown by general formula (I) of this invention can be manufactured by making the piperidine compound shown by the benzyl halide shown by formula (II), and formula (III) react under existence of a solvent and a base if needed.
[0047]What is necessary is just to usually consider it as an equimolar ratio or its neighborhood in this reaction, although the using rate in particular with the benzyl halide shown by the piperidine compound shown by formula (III) and formula (II) is not restricted but it can choose from the wide range suitably.
[0048]As a solvent used for this reaction, for example Aromatic hydrocarbon, such as benzene, toluene, xylene, or chlorobenzene. Fatty alcohol, such as methyl alcohol, ethyl alcohol, or tert-butyl alcohol. Ester species, such as nitril, such as acetonitrile or propionitrile, ethyl acetate, or ethyl propionate, Ether, such as diethylether, a tetrahydrofuran, or dioxane, Ketone, such as acetone or methyl ethyl ketone, a methylene chloride, Halogenated hydrocarbon, such as a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic hydrocarbon, such as pentane, hexane, heptane, or cyclohexane. Amide, such as N.N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, or 1,3-dimethyl-2-imidazolidinone. Dimethyl sulfoxide, pyridine, acetic acid, or water can be used, or these mixed solvents can also be used. Preferably, they are N.N-dimethylformamide, dimethyl sulfoxide, chloroform, acetone, a tetrahydrofuran, or acetonitrile.
[0049]In this reaction, although a base is not necessarily needed, it may carry out under existence of a base. As a base used, for example Triethylamine, diisopropylethylamine, Organic tertiary amines, such as tributylamine, 4-dimethylaminopyridine, pyridine, 1,4-diazabicyclo [2, 2, 2] octane or 1, and 8-diazabicyclo [5, 4, 0]-7-undecene. Alkaline metal alkoxides, such as sodium methoxide, a sodium ethoxide, or potassium tert-butoxide. Alkali metal carbonate, such as sodium carbonate, potassium carbonate, sodium bicarbonate, or potassium bicarbonate. Although hydrogenation alkaline metals, such as alkali metal hydroxide, such as sodium hydroxide or a potassium hydrate, sodium hydride, or potassium hydride, can be mentioned, They are triethylamine, diisopropylethylamine, pyridine, sodium carbonate, or potassium carbonate preferably. What is necessary is just to consider it as the amount of stoichiometries usually more superfluous than the amount of stoichiometries, or it then good, and desirable or quantity in which about 1 to 5 times is more superfluous than it, although the amount in particular of the above-mentioned base used is not restricted but it can choose from the wide range suitably. When using organic bases, such as triethylamine and pyridine, these can be used for an overlarge and it can also be considered as a solvent.
[0050]In this reaction, reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is -10 ** - 80 ** preferably.
[0051]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 30 minutes - 24 hours.
[0052]The compound of formula (I) obtained by the reaction process (A) is extracted by the usual post-processing out of a reaction solution. For example, it is obtained by adding an extracting solvent and water, such as toluene, ethyl acetate, or chloroform, to a reaction solution, and distilling off a solvent after rinsing. If the compound of obtained formula (I) is necessary, operation of column chromatography, recrystallization, etc. can refine it.
[0053]In a reaction process (A), the piperidine compound shown by formula (III) used as a raw material is a publicly known compound, for example, can be easily compounded in accordance with the method of a statement in a U.S. Pat. No. 5569664 specification, the No. Patent Publication Heisei 11-509524 gazette, etc. The example of concrete manufacture was shown in the after-mentioned examples 5 and 6 of reference manufacture.
[0054]The benzyl halide shown by formula (II) can be manufactured as the compound of the formula (IIa) in the after-mentioned reaction process (C), and a compound of the formula (IIb) of a reaction process (F), for example.
[0055]The piperidine derivative N-oxide object of the general formula (Ia) by this invention and the piperidine derivative salt of a general formula (Ib) can be manufactured by the method of the following reaction process (B), for example.
[0056]

反応工程(B)



(R_1, R_2, R_3, X, and Y show the same meaning as the above among a formula, and A−H shows an adduct.)

That is, in a reaction process (B), the piperidine derivative N−oxide object shown by the general formula (Ia) of this invention is manufactured by making the piperidine derivative and oxidizer which are shown by formula (I) react under existence of a catalyst a solvent and if needed.

[0057]As a solvent used for this reaction, for example Aromatic hydrocarbon, such as benzene, toluene, xylene, or chlorobenzene. Ester species, such as ethyl acetate or ethyl propionate, diethylether, Ketone, such as ether, such as a tetrahydrofuran or dioxane, acetone, or methyl ethyl ketone. Halogenated hydrocarbon, such as a methylene chloride, a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic hydrocarbon, such as pentane, hexane, heptane, or cyclohexane. Amide, such as N,N−dimethylformamide, N,N−dimethylacetamide, N−methyl−2−pyrrolidone, or 1,3−dimethyl−2−imidazolidinone, dimethyl sulfoxide, etc. can be used, or these mixed solvents can also be used. Preferably, they are chloroform or a methylene chloride.

[0058]As an oxidizer used in this reaction, m−chloroperbenzoic acid, hydrogen peroxide, fault iodine acid sodium, hypochlorous acid tert−butyl, or sodium hypochlorite can be used, and it is m−chloroperbenzoic acid preferably, for example.

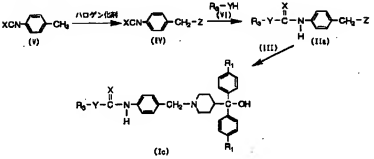
[0059]What is necessary is just to consider it as the amount of stoichiometries usually more superfluous than the amount of stoichiometries, or it then good, and desirable or quantity in

which about 1 to 3 times is more superfluous than it, although the amount in particular of the above-mentioned oxidizer used is not restricted but it can choose from the wide range suitably.
[0060]As a catalyst which can be used if needed, they are sodium tungstate or ammonium molybdate, for example.
[0061]Although the amount in particular of the catalyst used is not restricted but can be suitably chosen from the wide range, for what is necessary just to be usually 0.0001-1 times the amount of the amount of stoichiometries, and is just about 0.01-1 times the amount of the amount of stoichiometries preferably.
[0062]In this reaction, reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is -10 ** - 80 ** preferably.
[0063]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 10 minutes - 24 hours.
[0064]The compound of the general formula (Ia) obtained by the reaction process (B) is extracted by the usual post-processing out of a reaction solution. For example, it is obtained by adding an extracting solvent and water, such as toluene, ethyl acetate, or chloroform, to a reaction solution, and distilling off a solvent after rinsing. If the compound of the obtained general formula (Ia) is necessary, operation of column chromatography, recrystallization, etc. can refine it.
[0065]In a reaction process (B), the piperidine derivative salt shown by the formula (Ib) of this invention is manufactured by making the adduct shown by the piperidine derivative shown by formula (I), and A-H react under solvent existence if needed.
[0066]As a solvent used for this reaction, for example Aromatic hydrocarbon, such as benzene, toluene, xylene, or chlorobenzene. Fatty alcohol, such as methyl alcohol, ethyl alcohol, or tert-butyl alcohol. Ester species, such as nitril, such as acetonitrile or propionitrile, ethyl acetate, or ethyl propionate, Ether, such as diethylether, diisopropyl ether, a tetrahydrofuran, or dioxane, Ketone, such as acetone or methyl ethyl ketone, a methylene chloride, Halogenated hydrocarbon, such as a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic hydrocarbon, such as pentane, hexane, heptane, or cyclohexane. Amide, such as N,N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, or 1,3-dimethyl-2-imidazolidinone. Dimethyl sulfoxide, pyridine, acetic acid, or water can be used, or these mixed solvents can also be used. Preferably, they are diethylether, hexane, toluene, acetone, a tetrahydrofuran, or ethanol.
[0067]As a compound shown by A-H, for example Chloride, sulfuric acid, nitric acid, phosphoric acid, Inorganic acid, such as boric acid, acetic acid, propionic acid, oxalic acid, succinic acid, Organic acid, such as malonic acid, boletic acid, maleic acid, phthalic acid, salicylic acid, and D-glucuronic acid. Alcohols, water, etc., such as sulfonic acid, such as methanesulfonic acid, ethane sulfonic acid, 2-hydroxyethane sulfonic acid, and p-toluenesulfonic acid, methanol, ethanol, and ethylene glycol, can be used. Preferably, they are chloride, ethane sulfonic acid, 2-hydroxyethane sulfonic acid, ethanol, water, etc.
[0068]It should just presuppose that it is superfluous and what is necessary is just to usually desirable usually let it be the amount of stoichiometries, or the quantity in which about 1 to 3 times is more superfluous than it rather than the amount of stoichiometries, or it, although the amount in particular of the above-mentioned A-H used is not restricted but can be suitably chosen from the wide range.
[0069]In this reaction, reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is -10 ** - 80 ** preferably.
[0070]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 10 minutes - 24 hours.
[0071]The compound of the general formula (Ib) obtained by the reaction process (B) is extracted by the usual post-processing out of a reaction solution. For example, it is obtained by isolating a crystal by filtration operation in a precipitated crystal, or distilling off a reactional solvent. If the compound of the obtained general formula (Ib) is necessary, operation of column chromatography, recrystallization, etc. can refine it.
[0072]In the piperidine derivative shown by formula (I), R_2 can manufacture the piperidine

derivative shown by the formula (Ic) which is a hydrogen atom by the method of the following reaction process (C), for example.

[0073]

反応工程(C)(R_9=H)



(R_1, R_3, X, and Y show the same meaning as the above among a formula, and Z shows a halogen atom.)

That is, in a reaction process (C), the benzyl halide shown by formula (IV) can be manufactured by making the tolyl isocyanate or tolyl isothiocyanate shown by formula (V) react to a halogenating agent under existence of a catalyst first a solvent and if needed.

[0074]As a solvent used for this reaction, for example Aromatic hydrocarbon, such as benzene, toluene, xylene, or chlorobenzene. Fatty alcohol, such as methyl alcohol, ethyl alcohol, or tert-butyl alcohol. Ester species, such as nitril, such as acetonitrile or propionitrile, ethyl acetate, or ethyl propionate, Ether, such as diethylether, a tetrahydrofuran, or dioxane, Ketone, such as acetone or methyl ethyl ketone, a methylene chloride, Halogenated hydrocarbon, such as a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic hydrocarbon, such as pentane, hexane, heptane, or cyclohexane. Amide, such as N,N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, or 1,3-dimethyl-2-imidazolidinone, or dimethyl sulfoxide can be used, or these mixed solvents can also be used. Preferably, it is a carbon tetrachloride.

[0075]As a halogenating agent used for this reaction, it is halogen, such as imide, such as N-chlorosuccinimid or N-bromosuccinimid, chlorine, or bromine, etc., for example.

[0076]It should just presuppose that it is superfluous and what is necessary is just to usually desirable usually let it be the amount of stoichiometries, or the quantity in which about 1 to 3 times is more superfluous than it rather than the amount of stoichiometries, or it, although the amount in particular of the above-mentioned halogenating agent used is not restricted but can be suitably chosen from the wide range.

[0077]As a catalyst used for this reaction, when imide is used as a halogenating agent, peroxides, such as nitril, such as 2,2'-azobis (isobutyronitrile), or benzoyl peroxide, etc. can be used, for example.

[0078]What is necessary is for what is necessary to be just to consider it as 0.0001-1 times the amount of the amount of stoichiometries, and just to be about 0.01-1 times the amount of the amount of stoichiometries preferably as amount of the catalyst used.

[0079]When halogen is used as a halogenating agent, it can be made to react under an optical exposure.

[0080]In this reaction, reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is -10 ** - 80 ** preferably.

[0081]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 30 minutes - 24 hours.

[0082]Thus, the compound of obtained formula (IV) is extracted by the usual post-processing out of a reaction solution. For example, it is obtained by adding an extracting solvent and water, such as a carbon tetrachloride or chloroform, to a reaction solution, and distilling off a solvent after rinsing. If the compound of obtained formula (IV) is necessary, operation of column

chromatography, recrystallization, etc. can refine it.
[0083]Next, in a reaction process (C), the Cava mate to whom it is shown by a formula (IIa) can be manufactured by making the compound of formula (IV) react to alcohol or mercaptan which is formula (VI) and is shown under existence of a solvent and a catalyst if needed.
[0084]In this reaction, although a solvent is not necessarily needed, it may carry out under existence of a solvent. As a solvent used, for example Aromatic hydrocarbon, such as benzene, toluene, xylene, or chlorobenzene. Nitril shown by formula (VI), such as alcohol or mercaptans, acetonitrile, or propionitrile. Ester species, such as ethyl acetate or ethyl propionate, diethylether, Ketone, such as ether, such as a tetrahydrofuran or dioxane, acetone, or methyl ethyl ketone. Halogenated hydrocarbon, such as a methylene chloride, a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic hydrocarbon, such as pentane, hexane, heptane, or cyclohexane. Amide, such as N,N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, or 1,3-dimethyl-2-imidazolidinone, or dimethyl sulfoxide can be used, or these mixed solvents can also be used. Preferably, they are N,N-dimethylformamide, acetonitrile, chloroform, etc.
[0085]What is necessary is just to usually consider it as an equimolar ratio or its neighborhood in this reaction, although the using rate in particular of the compound of formula (IV) and the compound of formula (VI) is not restricted but it can choose from the wide range suitably. The compound of formula (VI) can be used for an overlarge, and it can also be considered as a solvent.
[0086]In this reaction, although a catalyst is not necessarily needed, it may carry out under existence of a catalyst. As a catalyst used, for example Triethylamine, diisopropylethylamine, Although there are organic tertiary amines, such as tributylamine, 4-dimethylaminopyridine, pyridine, 1,4-diazabicyclo [2, 2, 2] octane or 1, and 8-diazabicyclo [5, 4, 0]-7-undecene, Preferably, they are triethylamine, diisopropylethylamine, pyridine, etc.
[0087]What is necessary is for what is necessary to be just to consider it as 0.0001-1 times the amount of the amount of stoichiometries, and just to be about 0.01-1 times the amount of the amount of stoichiometries preferably as amount of the catalyst used.
[0088]Reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is -10 ** - 80 ** preferably.
[0089]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 30 minutes - 24 hours.
[0090]Thus, the compound of the obtained formula (IIa) is extracted by the usual post-processing out of a reaction solution. For example, it is obtained by adding an extracting solvent and water, such as toluene, ethyl acetate, or chloroform, to a reaction solution, and distilling off a solvent after rinsing. If the compound of the obtained formula (IIa) is necessary, operation of column chromatography, recrystallization, etc. can refine it.
[0091]In the final process of a reaction process (C), the piperidine derivative shown by the general formula (Ic) of this invention can be manufactured by making the piperidine compound shown by the benzyl halide shown by a formula (IIa), and formula (III) react under existence of a solvent and a base if needed.
[0092]About reaction operation, it can carry out by the same method as a reaction process (A).
[0093]In a reaction process (C), the compound of a formula (Ic) can be manufactured with a one pot system from the compound of formula (IV), without isolating the compound of the formula (IIa) which is an intermediate.
[0094]In this reaction, the Cava mate intermediate shown by a formula (IIa) can be manufactured by making the compound of formula (IV) react to alcohol or mercaptan shown by formula (VI) under existence of a solvent and a catalyst if needed.
[0095]In this reaction, although a solvent is not necessarily needed, it may carry out under existence of a solvent. As a solvent used, for example Aromatic hydrocarbon, such as benzene, toluene, xylene, or chlorobenzene. Ester species, such as nitril, such as acetonitrile or propionitrile, ethyl acetate, or ethyl propionate, Ether, such as diethylether, a tetrahydrofuran, or dioxane, Ketone, such as acetone or methyl ethyl ketone, a methylene chloride, Halogenated hydrocarbon, such as a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic

hydrocarbon, such as pentane, hexane, heptane, or cyclohexane. Amide, such as N,N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, or 1,3-dimethyl-2-imidazolidinone, or dimethyl sulfoxide can be used, or these mixed solvents can also be used. Preferably, they are N,N-dimethylformamide, acetonitrile, chloroform, etc.

[0096]What is necessary is just to usually consider it as an equimolar ratio or its neighborhood in this reaction, although the using rate in particular of the compound of formula (IV) and the compound of formula (VI) is not restricted but it can choose from the wide range suitably.

[0097]In this reaction, although a catalyst is not necessarily needed, it may carry out under existence of a catalyst. As a catalyst used, for example Triethylamine, diisopropylethylamine, Although there are organic tertiary amines, such as tributylamine, 4-dimethylaminopyridine, pyridine, 1,4-diazabicyclo [2, 2, 2] octane or 1, and 8-diazabicyclo [5, 4, 0]-7-undecene, Preferably, they are triethylamine, diisopropylethylamine, pyridine, etc.

[0098]What is necessary is for what is necessary to be just to consider it as 0.0001-1 times the amount of the amount of stoichiometries, and just to be about 0.01-1 times the amount of the amount of stoichiometries preferably as amount of the catalyst used.

[0099]Reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is -10 ** - 80 ** preferably.

[0100]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 30 minutes - 24 hours.

[0101]Next, the piperidine derivative shown by formula (I) can be manufactured by making the piperidine compound shown by the reaction mixture containing the compound of the obtained formula (IIa), and formula (III) react under existence of a solvent and a base if needed.

[0102]About reaction operation, it can carry out by the same method as a reaction process (A).

[0103]In the field of organic chemistry, the tolyl isocyanate or tolyl isothiocyanate shown by formula (V) which is used by a reaction process (C), and which is a starting material is the compound known well, for example, can be obtained as a reagent from Tokyo Kasei Kogyo Co., Ltd.

[0104]The benzyl halide which is used by a reaction process (C) and which is shown by formula (IV), It is a publicly known compound, for example, is a journal. OBU Heterocyclic Chemistry (Journal of Heterocyclic Chemistry), The 31st volume, the 457th page - the 479th page (1994), the 376th page - the 377th page (1978) of synthesis (Synthesis), KOREKUSHONOBU Czechoslovakia Slovak Chemical Communications (Collection of Czechoslovak Chemical Communications), In accordance with the method of a statement, it can manufacture easily in the 55th volume, the 752nd page - the 760th page (1990), JP,51-19756,A, and the British patent public presentation No. 752931 gazette. The example of concrete manufacture was shown in the after-mentioned examples 1 and 4 of reference manufacture.

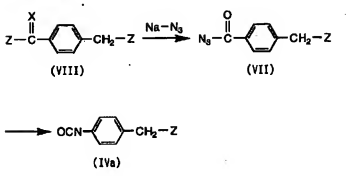
[0105]In the field of organic chemistry, alcohol or mercaptan which is used by a reaction process (C) and which is shown by formula (VI) is the compound known well, for example, can be obtained as a reagent from Tokyo Kasei Kogyo Co., Ltd.

[0106]The benzyl halide shown by a formula (IIa) could be manufactured as shown in the reaction process (C), but it showed the example of concrete manufacture further to the after-mentioned example 1 of reference manufacture.

[0107]In the compound of formula (IV) of a reaction process (C), the isocyanate compound in which especially X is shown by the formula (IVa) which is an oxygen atom can be manufactured also by the method of a reaction process (D), for example.

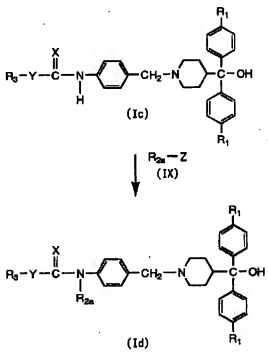
[0108]

反応工程(D)



(Z shows a halogen atom among a formula.)
That is, in a reaction process (D), the acid azide compound shown by formula (VII) can be manufactured by making the acid halide compound shown by formula (VIII) react to sodium azide under solvent existence first.
[0109]As a solvent used for this reaction, for example Aromatic hydrocarbon, such as benzene, toluene, xylene, or chlorobenzene. Fatty alcohol, such as methyl alcohol, ethyl alcohol, or tert-butyl alcohol. Ester species, such as nitril, such as acetonitrile or propionitrile, ethyl acetate, or ethyl propionate, Ether, such as diethylether, a tetrahydrofuran, or dioxane, Ketone, such as acetone or methyl ethyl ketone, a methylene chloride, Halogenated hydrocarbon, such as a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic hydrocarbon, such as pentane, hexane, heptane, or cyclohexane. Amide, such as N,N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, or 1,3-dimethyl-2-imidazolidinone. Dimethyl sulfoxide, pyridine, acetic acid, or water can be used, or these mixed solvents can also be used. Preferably, they are N,N-dimethylformamide, acetone, a tetrahydrofuran, diethylether, dioxane, etc.
[0110]It should just presuppose that it is superfluous and what is necessary is just to usually desirable usually let it be the amount of stoichiometries, or the quantity in which about 1 to 3 times is more superfluous than it rather than the amount of stoichiometries, or it, although the amount in particular of the sodium azide used is not restricted but can be suitably chosen from the wide range.
[0111]Reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is -10 ** - 80 ** preferably.
[0112]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 30 minutes - 24 hours.
[0113]Thus, the compound of obtained formula (VII) is extracted by the usual post-processing out of a reaction solution. For example, it is obtained by adding an extracting solvent and water, such as toluene and diethylether, to a reaction solution, and distilling off a solvent after rinsing. If the compound of obtained formula (VII) is necessary, operation of column chromatography, recrystallization, etc. can refine it.
[0114]The compound of a formula (IVa) can be manufactured [in / next / a reaction process (D)] by carrying out the pyrogenetic reaction of the compound of formula (VII) under existence of a solvent if needed.
[0115]As a solvent used for this reaction, for example Aromatic hydrocarbon, such as benzene, toluene, xylene, or chlorobenzene. Fatty alcohol, such as methyl alcohol, ethyl alcohol, or tert-butyl alcohol. Ester species, such as nitril, such as acetonitrile or propionitrile, ethyl acetate, or ethyl propionate, Ether, such as diethylether, a tetrahydrofuran, or dioxane, Ketone, such as acetone or methyl ethyl ketone, a methylene chloride, Halogenated hydrocarbon, such as a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic hydrocarbon, such as pentane, hexane, heptane, or cyclohexane. Amide, such as N.N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, or 1,3-dimethyl-2-imidazolidinone. Dimethyl sulfoxide, pyridine, acetic acid, or water can be used, or these mixed solvents can also be used. Preferably, they are benzene, toluene, etc.

[0116]Reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is 60 ** - 120 ** preferably.
[0117]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 30 minutes - 24 hours.
[0118]In a reaction process (D), the compound of a formula (IVa) can also be manufactured by a one pot from the compound of formula (VIII), without isolating the compound of formula (VII) which is an intermediate.
[0119]In a reaction process (D), in the field of organic chemistry, the acid halide compound shown by formula (VIII) is a compound known well, for example, can be obtained as a reagent from Tokyo Kasei Kogyo Co., Ltd.
[0120]The manufacture of the isocyanate compound shown by a formula (IVa) by a reaction process (D) is an INOGA nick, for example. In accordance with the method of chemistry (Inorganic Chemistry), the 23rd volume, and a statement (the 961st page - the 969th page (1984)), it can carry out easily. The example of concrete manufacture was shown in the after-mentioned example 3 of reference manufacture.
[0121]In the piperidine derivative shown by formula (I), when R_2 is not a hydrogen atom, the piperidine derivative shown by a formula (Id) can be manufactured by the method of the following reaction process (E), for example.
[0122]
反応工程(E)



(Among a formula, R_1, R_3, X, and Y show the same meaning as the above, R_2a shows R_2 of the same meaning as the above except a hydrogen atom, and Z shows a halogen atom.)
That is, in a reaction process (E), the piperidine derivative shown by a formula (Id) can be manufactured by making the compound of a formula (Ic) react to the halide compound which is formula (IX) and is shown under existence of a solvent and a base if needed.
[0123]As a solvent used for this reaction, for example Aromatic hydrocarbon, such as benzene, toluene, xylene, or chlorobenzene. Fatty alcohol, such as methyl alcohol, ethyl alcohol, or tert-butyl alcohol. Ester species, such as nitril, such as acetonitrile or propionitrile, ethyl acetate, or ethyl propionate, Ether, such as diethylether, a tetrahydrofuran, or dioxane, Ketone, such as acetone or methyl ethyl ketone, a methylene chloride, Halogenated hydrocarbon, such as a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic hydrocarbon, such as pentane,

hexane, heptane, or cyclohexane. Amide, such as N,N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, or 1,3-dimethyl-2-imidazolidinone. Dimethyl sulfoxide, pyridine, acetic acid, or water can be used, or these mixed solvents can also be used. Preferably, they are toluene, acetonitrile, chloroform, N,N-dimethylformamide, etc.

[0124]In this reaction, although a base is not necessarily needed, it may carry out under existence of a base. As a base used, for example Triethylamine, diisopropylethylamine, Organic tertiary amines, such as tributylamine, 4-dimethylaminopyridine, pyridine, 1,4-diazabicyclo [2, 2, 2] octane or 1, and 8-diazabicyclo [5, 4, 0]-7-undecene. Alkaline metal alkoxides, such as sodium methoxide, a sodium ethoxide, or potassium tert-butoxide. Alkali metal carbonate, such as sodium carbonate, potassium carbonate, sodium bicarbonate, or potassium bicarbonate. Although hydrogenation alkaline metals, such as alkali metal hydroxide, such as sodium hydroxide or a potassium hydrate, sodium hydride, or potassium hydride, can be raised, they are triethylamine, sodium hydride, sodium carbonate, or potassium carbonate preferably. What is necessary is just to consider it as the amount of stoichiometries usually more superfluous than the amount of stoichiometries, or it then good, and desirable or quantity in which about 1 to 5 times is more superfluous than it, although the amount in particular of the above-mentioned base used is not restricted but it can choose from the wide range suitably. When using organic bases, such as triethylamine and pyridine, these can be used for an overlarge and it can also be considered as a solvent.

[0125]What is necessary is just to consider it as the amount of stoichiometries usually more superfluous than the amount of stoichiometries, or it then good, and desirable or quantity in which about 1 to 5 times is more superfluous than it, although the amount in particular of the compound used of formula (IX) used for this reaction is not restricted but it can choose from the wide range suitably.

[0126]In this reaction, reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is -10 ** - 80 ** preferably.

[0127]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 30 minutes - 24 hours.

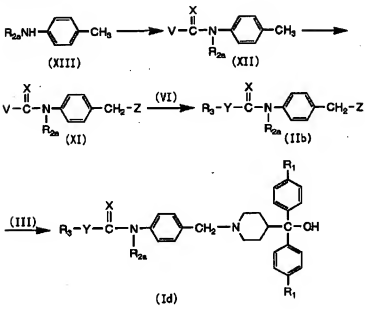
[0128]The compound of the formula (Id) obtained by the reaction process (E) is extracted by the usual post-processing out of a reaction solution. For example, it is obtained by adding an extracting solvent and water, such as toluene, ethyl acetate, or chloroform, to a reaction solution, and distilling off a solvent after rinsing. If the compound of the obtained formula (Id) is necessary, operation of column chromatography, recrystallization, etc. can refine it.

[0129]In a reaction process (E), in the field of organic chemistry, the halide compound which is formula (IX) and is shown is a compound known well, for example, can be obtained as a reagent from Tokyo Kasei Kogyo Co., Ltd.

[0130]The compound of a formula (Id) can be manufactured also by the method of a reaction process (F) again.

[0131]

反応工程(F)



(XIII) (XII) (XI) (IIb) (Id)

(R_1, R_3, X, and Y carry out the same meaning as the above among a formula, R_2a shows R_2 of the same meaning as the above except a hydrogen atom, and Z and V show a halogen atom.) That is, in a reaction process (F), the carbamoyl halide shown by formula (XII) can be first manufactured by making the compound of formula (XIII) react to a carbamoylation agent under existence of a solvent if needed.

[0132]As a solvent used for this reaction, for example Aromatic hydrocarbon, such as benzene, toluene, xylene, or chlorobenzene. Fatty alcohol, such as methyl alcohol, ethyl alcohol, or tert-butyl alcohol. Ester species, such as nitril, such as acetonitrile or propionitrile, ethyl acetate, or ethyl propionate, Ether, such as diethylether, a tetrahydrofuran, or dioxane, Ketone, such as acetone or methyl ethyl ketone, a methylene chloride, Halogenated hydrocarbon, such as a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic hydrocarbon, such as pentane, hexane, heptane, or cyclohexane. Amide, such as N,N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, or 1,3-dimethyl-2-imidazolidinone, dimethyl sulfoxide, pyridine, or acetic acid can be used, or these mixed solvents can also be used. Preferably, it is toluene.

[0133]As a carbamoylation agent, they are phosgene and its polymer, thiophosgene, etc. What is necessary is just to consider it as the amount of stoichiometries usually more superfluous than the amount of stoichiometries, or it then good, and desirable or quantity in which about 1 to 3 times is more superfluous than it, although the amount used in particular is not restricted but it can choose from the wide range suitably.

[0134]Reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is 20 ** - the flowing-back temperature in the system of reaction preferably.

[0135]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 30 minutes - 24 hours.

[0136]The compound of obtained formula (XII) is extracted by the usual post-processing out of a reaction solution. For example, it is obtained by distilling off a solvent. If the compound of obtained formula (XII) is necessary, operation of column chromatography, recrystallization, etc. can refine it.

[0137]Next, the benzyl halide shown by formula (XI) can be manufactured by making the compound of formula (XII) react to a halogenating agent under existence of a catalyst a solvent and if needed.

[0138]As a solvent used for this reaction, for example Aromatic hydrocarbon, such as benzene,

toluene, xylene, or chlorobenzene. Fatty alcohol, such as methyl alcohol, ethyl alcohol, or tert-butyl alcohol. Ester species, such as nitril, such as acetonitrile or propionitrile, ethyl acetate, or ethyl propionate, Ether, such as diethylether, a tetrahydrofuran, or dioxane, Ketone, such as acetone or methyl ethyl ketone, a methylene chloride, Halogenated hydrocarbon, such as a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic hydrocarbon, such as pentane, hexane, heptane, or cyclohexane. Amide, such as N.N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, or 1,3-dimethyl-2-imidazolidinone, or dimethyl sulfoxide can be used, or these mixed solvents can also be used. Preferably, it is a carbon tetrachloride.

[0139]As a halogenating agent used for this reaction, it is halogen, such as imide, such as N-chlorosuccinimid or N-bromosuccinimid, chlorine, or bromine, etc., for example.

[0140]It should just presuppose that it is superfluous and what is necessary is just to usually desirable usually let it be the amount of stoichiometries, or the quantity in which about 1 to 3 times is more superfluous than it rather than the amount of stoichiometries, or it, although the amount in particular of the above-mentioned halogenating agent used is not restricted but can be suitably chosen from the wide range.

[0141]As a catalyst used for this reaction, when imide is used as a halogenating agent, peroxides, such as nitril, such as 2,2'-azobis (isobutyronitrile), or benzoyl peroxide, etc. can be used, for example.

[0142]What is necessary is for what is necessary to be just to consider it as 0.0001-1 times the amount of the amount of stoichiometries, and just to be about 0.01-1 times the amount of the amount of stoichiometries preferably as amount of the catalyst used.

[0143]When halogen is used as a halogenating agent, it can be made to react under an optical exposure.

[0144]In this reaction, reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is -10 ** - 80 ** preferably.

[0145]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 30 minutes - 24 hours.

[0146]Thus, the compound of obtained formula (XI) is extracted by the usual post-processing out of a reaction solution. For example, it is obtained by adding an extracting solvent and water, such as a carbon tetrachloride or chloroform, to a reaction solution, and distilling off a solvent after rinsing. If the compound of obtained formula (XI) is necessary, operation of column chromatography, recrystallization, etc. can refine it.

[0147]Next, the Cava mate to whom it is shown by a formula (IIb) can be manufactured by making the compound of formula (XI) react to alcohol or mercaptan shown by formula (VI) under existence of a solvent and a base if needed.

[0148]In this reaction, as a solvent used, for example Benzene, toluene, Aromatic hydrocarbon, such as xylene or chlorobenzene, the alcohol shown by formula (VI), or mercaptans, Ester species, such as nitril, such as acetonitrile or propionitrile, ethyl acetate, or ethyl propionate, Ether, such as diethylether, a tetrahydrofuran, or dioxane, Ketone, such as acetone or methyl ethyl ketone, a methylene chloride, Halogenated hydrocarbon, such as a dichloroethane, chloroform, or a carbon tetrachloride. Aliphatic hydrocarbon, such as pentane, hexane, heptane, or cyclohexane. Amide, such as N.N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, or 1,3-dimethyl-2-imidazolidinone, dimethyl sulfoxide, or pyridine can be used, or these mixed solvents can also be used. Preferably, they are N.N-dimethylformamide, acetonitrile, a tetrahydrofuran, etc.

[0149]What is necessary is just to usually consider it as an equimolar ratio or its neighborhood in this reaction, although the using rate in particular of the compound of formula (XI) and the compound of formula (VI) is not restricted but it can choose from the wide range suitably. The compound of formula (VI) can be used for an overlarge, and it can also be considered as a solvent.

[0150]In this reaction, as a base used, for example Triethylamine, Diisopropylethylamine, tributylamine, 4-dimethylaminopyridine, Organic tertiary amines, such as pyridine, 1,4-diazabicyclo [2, 2, 2] octane or 1, and 8-diazabicyclo [5, 4, 0]-7-undecene. Alkaline metal

alkoxides, such as sodium methoxide, a sodium ethoxide, or potassium tert-butoxide. Alkali metal carbonate, such as sodium carbonate, potassium carbonate, sodium bicarbonate, or potassium bicarbonate. Although hydrogenation alkaline metals, such as alkali metal hydroxide, such as sodium hydroxide or a potassium hydrate, sodium hydride, or potassium hydride, can be raised, they are potassium carbonate and sodium hydride preferably. What is necessary is just to consider it as the amount of stoichiometries usually more superfluous than the amount of stoichiometries, or it then good, and desirable or quantity in which about 1 to 5 times is more superfluous than it, although the amount in particular of the above-mentioned base used is not restricted but it can choose from the wide range suitably.

[0151]In this reaction, reaction temperature is performed at -30 ** - the flowing-back temperature in the system of reaction, and is 20 ** - the flowing-back temperature in the system of reaction preferably.

[0152]Although reaction time changes with reaction temperature or reactional substrates, a reaction usually completes it in 30 minutes - 24 hours.

[0153]The compound of the obtained formula (IIb) is extracted by the usual post-processing out of a reaction solution. For example, it is obtained by adding an extracting solvent and water, such as toluene, ethyl acetate, or chloroform, to a reaction solution, and distilling off a solvent after rinsing. If the compound of the obtained formula (IIb) is necessary, operation of column chromatography, recrystallization, etc. can refine it.

[0154]In the final process of a reaction process (F), the piperidine derivative shown by the formula (Id) of this invention can be manufactured by making the compound of a formula (IIb), and the compound of formula (III) react under existence of a solvent and a base if needed.

[0155]About reaction operation, it can carry out by the same method as a reaction process (A).

[0156]Formula (XII) in a reaction process (F), (XI), and the example of concrete manufacture of the compound of (IIb) were shown in the after-mentioned example 2 of reference manufacture.

[0157]Next, the method of general pharmaceutical-preparation-izing of the piperidine derivative of general formula (I) by this invention is explained in detail.

[0158]It faces using this invention compound as an active principle of an insecticide, Although it may use by this invention compound itself, the carrier, the surface-active agent, and the other adjuvants which are generally as an agricultural-chemicals adjuvant used to pharmaceutical-preparation-izing are blended, Medicine can be manufactured in various gestalten, such as an emulsion, suspension, powder material, a granule, a tablet, wettable powder, water soluble powders, liquids and solutions, a floor bull agent, granulation wettable powder, aerosols, a paste agent, oils, and an opacifier. These blending ratios are usually ten to agricultural-chemicals adjuvant 99.9 weight sections in 0.1 to active principle 90 weight section.

[0159]As a carrier which can be used on the occasion of pharmaceutical-preparation-izing, if regularly used by the drugs for plantation arts, either a solid support or a liquid carrier can be used, and it will not be limited to a specific thing.

[0160]As such an example, for example as a solid support, For example, mineral powder, such as animals-and-plants nature powder, such as starch, activated carbon, soybean flour, wheat flour, wood flour, fish meal, and powdered milk, talc, kaolin, bentonite, zeolite, diatomaceous earth, white carbon, clay, alumina, calcium carbonate, potassium chloride, and ammonium sulfate, is mentioned.

[0161]As a liquid carrier, for example Alcohols; cyclohexanones, such as water; isopropyl alcohol and ethylene glycol, Ketone, such as methyl ethyl ketone; Propylene glycol monomethyl ether, Ether, such as diethylene-glycol mono-n-butyl ether; Kerosene, Aliphatic hydrocarbon, such as gas oil; Xylene, trimethyl benzene, tetramethyl benzene, Ester species, such as glycerol ester of amide; fatty acid, such as aromatic hydrocarbon; N-methyl-2-pyrrolidone, such as methylnaphthalene and solvent naphtha; vegetable oil, such as soybean oil and rapeseed oil, is mentioned. These carriers can use two or more sorts together.

[0162]As a surface-active agent which can be used on the occasion of pharmaceutical-preparation-izing, there are a nonionic surfactant, an anionic detergent, a cationic surfactant, an ampholytic surface active agent, etc., and the following can specifically be used.

[0163]As an example of a nonionic surfactant, for example Polyoxyethylene alkyl ether,

Polyoxyethylene alkyl aryl ether, polyoxyethylene styryl phenyl ether, Polyoxyethylene alkyl ester, a polyoxyethylene sorbitan alkylate, Polyoxyethylene phenyl ether polymer, polyoxyethylene alkylene aryl phenyl ether, polyoxyethylene alkylene glycol, a polyoxyethylene polyoxypropylene blockpolymer, etc. are mentioned.
[0164]As an example of an anionic detergent, for example A ligninsulfonic acid salt, Alkylaryl sulfonates, dialkyl sulfosuccinate, polyoxyethylene alkyl aryl ethersulfate, alkylnaphthalenesulfonate, polyoxyethylene styryl phenyl ether sulfate, etc. are mentioned.
[0165]As an example of a cationic surfactant, an alkylamine salt etc. are mentioned, for example.

[0166]As an example of an ampholytic surface active agent, a quarternary-ammonium-salt alkyl betaine, amine oxide, etc. are mentioned, for example.
[0167]The surface-active agent which can be used on the occasion of pharmaceutical-preparation-izing is not limited to these, and can also use together these two or more sorts.
[0168]Although a binder, a thickener, an adhesive agent, a preservation-from-decay antifungal agent, a solvent, the stabilizing agent of an agrochemical active ingredient, an antioxidant, ultraviolet inhibitor, a crystal deposit inhibitor, a defoaming agent, a physical-properties improver, colorant, etc. may be respectively added as other adjuvants if needed, it is not limited to the adjuvant illustrated here.
[0169]Although not limited especially as a binder, a thickener, and an adhesive agent, the following is mentioned, for example. Starch, dextrin, cellulose, methyl cellulose, ethyl cellulose, Carboxymethyl cellulose, hydroxyethyl cellulose, hydroxypropylcellulose, Hydroxypropylmethylcellulose, carboxymethyl starch, Pullulan, sodium alginate, ammonium alginate, propylene glycol alginate, Guar gum, locust bean gum, gum arabic, xanthan gum, gelatin, casein, polyvinyl alcohol, polyethylene oxide, a polyethylene glycol, ethylene propylene block polymer, sodium polyacrylate, a polyvinyl pyrrolidone, etc.
[0170]The pharmaceutical preparation of the insecticide of this invention obtained by a described method is used as follows.
[0171]That is, it can be used as it is, or can be diluted and used for prescribed concentration with diluents, such as water. Various pharmaceutical preparation containing this invention compound, or use of the dilution, Usually, the application method currently generally performed, i.e., spraying, soil use (for example, spraying, misting, atomizing, dusting, granule application, application on water surface, box use, etc.) (for example, mixing, douche, etc.), surface use (for example, spreading, dust coating, covering, etc.), immersion, poison bait, etc. can perform.
[0172]Although the amount of application in particular of the insecticide of this invention is not limited but it can choose from the wide range suitably according to various conditions, such as the amount of the kind of active principle concentration, the noxious insect for a gestalt of pharmaceutical preparation, or crops, the grade of the damage caused by a noxious insect, a use place, application method, a use stage, the drugs that carry out mixed use concomitant use, the manure used, etc., and a kind, usually, per [0.001-100] 100-m^2 — it is preferably used about 0.01-50g about g.
[0173]When diluting an emulsion, wettable powder, a floor bull agent, etc. with water and using them, about 0.1-1000 ppm of the use concentration is preferably used at about 1-500 ppm, but it is not limited to these. A granule, powder material, etc. are used with pharmaceutical preparation, without diluting.
[0174]Even when the pharmaceutical preparation of the insecticide of this invention is independent, an effective enough thing cannot be overemphasized, but it comes out to accept necessity, to use together and to use together with other manure, agricultural chemicals, for example, an insecticide, miticide, a nematicide, a germicide, an antivirotic, a attractant, a weed killer, a plants growth regulator, a synergist, etc.
[0175]The insecticide of this invention is applicable to prevention of the breeding and extermination of the following noxious insect, for example. However, application of the insecticide of this invention is not limited to these noxious insects.
[0176]As Hemiptera [Hemiptera], for example Nephotettix (Nephotettix cincticeps), Sogatella

furcifera (Sogatella furcifera), a rice brown planthopper (Nilaparvata lugens), A small brown planthopper (Laodelphax striatellus), Riptortus clavatus (Riptortus clavatus), a MINAMIAO bug (Nezara viridula), Pear Tingidae (Stephanitis nashi), an ONSHITSU white fly (Trialeurodes vaporariorum), An woolly aphis (Aphis gossypii), a green peach aphid (Myzus persicae), an Arrowhead scale (Unaspis yanonensis), etc.

[0177]As Lepidoptera [Lepidoptera], for example Phyllonorycter ringoniella (Phyllonorycterringoniella), A cabbage moth (Plutella xylostella), WATAMIGA (Promalactis inonisema), Apple Adoxophyes (Adoxophyes orana), tea HAMAKI (Homona magnanima), A soybean pod borer (Leguminivora glycinivorella), Cnaphalocrocis medinalis (Cnaphalocrocis medinalis), Chilo (Chilo supperessalis), Ostrinia furnacalis (Ostrinia furnacalis), A cabbage armyworm (Mamestra brassicae), Leucania (Pseudaletiaseparata), A tobacco cutworm (Spodoptera litura), rice TSUTOMUSHI (Parnaraguttata), A cabbage butterfly (Pieris rapae crucivora), HERIOCHISU (Heliothisspp.), a black cutworm (Agrotis ipsilon), Helicoverpa armigera (Helicoverpa armigera), etc.

[0178]As beetles [Coleoptera], for example A DOUGANE buoy buoy (Anomala cuprea), A Japanese beetle (Popillia japonica), a rice elephant beetle (Echinocnemus squameus), A rice Ms. elephant beetle (Lissorhoptrus oryzophilus), Rice DOROOIMUSHI (Oulema oryzae), a HIMEMARU carpet beetle (Anthrenus verbasci), A cadelle (Tenebroides mauritanicus), A rice weevil (Sitophilus zeamais), a NIJUUYAHOSHI ten tow (Epilachna vigintioctopunctata), Callosobruchus (Callosobruchus chinensis), Monochamus alternatus (Monochamus alternatus), Aulacophora femoralis (Aulacophora femoralis), JIABUROCHIKA (Diabrotica spp.), etc.

[0179]As Hymenoptera [Hymenoptera], they are KABURAHABACHI (Athalia rosae ruficornis), a RURICHUU range (Arge similis), etc., for example.

[0180]As Diptera [Diptera], for example Culex fatigans (Culex pipiens fatigans), SHIMAKA (Aedes spp.), a DAIZUSAYA gall midge (Asphondylia spp.), A seed-corn fly (Delia platura), a muscid (Musca domestica vicina), a melon fruit fly (Dacus cucurbitae), Agromyza oryzae (Agromyza oryzae), Kyn Valle (Lucilia spp.), etc.

[0181]As Siphonaptera [Aphaniptera], they are the Pulex irritans (Pulex irritans), an oriental rat flea (Xenopsylla cheopis), a dog flea (Ctenocephalides canis), etc., for example.

[0182]As Thysanoptera [Thysanoptera], for example Scirtothrips dorsalis (Scirtothrips dorsalis), A Welsh onion thrip (Thrips tabaci), a MINAMIKIIRO thrip (Thrips palmi), a rice thrip (Stenchaetothrips biformis), etc.

[0183]As Siphunculata [Anoplura], they are a body louse (Pediculs humanus corporis), the crab (Phthirius pubis), etc., for example.

[0184]As Psocoptera [Psocoptera], they are KOCHATATE (Trogium pulsatorium), HIRATACHATATE (Liposcelis bostrychophilus), etc., for example.

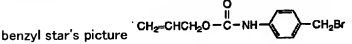
[0185]As Orthoptera [Orthoptera], for example A mole cricket (Gryllotalpa spp.), A locust (Locusta migratoria), Oxya japonica (Oxya yezoensis), Blattella germanica (Blattella germanica), Periplaneta fuliginosa (Periplaneta fuliginosa), etc.

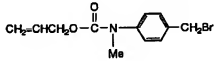
[0186]As a Isoptera noxious insect, they are Reticulitermes (Reticulitermes speratus), Coptotermes formosanus (Coptotermes formosanus), etc., for example.

[0187]As spider mites, for example A twospotted spider mite (Tetranychus urticae), KANZAWAHADANI (Tetranychus kanzawai), a citrus red mite (Panonychus citri), a European red mite (Panonychus ulmi), a mandarin orange rust mite (Aculops pelekassi), etc.

[0188]As vegetable parasitic Nematoda, they are a sweet potato root-knot nematode (Meloidogyne incognita), a meadow nematode (Pratylenchus spp.), DAIZUSHISUTOSENCHUU (Heterodera glycines), etc., for example.

[0189]As a noxious animal, an unpleasant animal, a medically important insect, and a parasite, for example In addition, a SUKUMI apple guy (Pomacea canaliculata), Gastropoda (Gastropoda), such as a slug (Incilaria sp.) and Achatina (Achatina fulica). DANGOMUSHI (Armadillidium spp.), a sow bug, Biting lice, such as Isopoda (Isopoda), such as centipede, and Trichodectes spp., Animal parasitic Acari, such as Cimex lectularius, such as Cimex spp., Boophilus microplus (Boophilus microplus), and Haemaphysalis longicornis (Haemaphysalis longicornis), Epidermoptidae, etc. can be mentioned.

[0190]
[Example]The examples 1-6 of reference manufacture and Examples 1-13 are given to below, and the example of manufacture of the compound of general formula (I) by this invention is explained.
[0191]Manufacture of an example of reference manufacture 14-(2-propenyloxy carbonylamino) benzyl star's picture 

a) 2.67g (20mmol) of 4-tolyl isocyanates and 25 ml of carbon tetrachlorides were put in and heated in 100-ml 4 mouth flask equipped with the thermometer which can be measured to the manufacture agitating equipment of 4-(bromomethyl) phenylisocyanate, a reflux condenser, and 100 **. Heating stirring was further carried out at 70 ** for 1.5 hours [divide into 0.33 g (2mmol) of 2,2'-azobis (isobutyronitrile) at 40 **, and / ** / 70 / succinimid / 3.56 g (20mmol) of / N-bromo/ times / 3]. Chloroform and chilled water were added and extracted in the reaction mixture. The chloroform layer was dried with anhydrous sodium sulfate, the solvent was distilled off under vacuum pump decompression, and 4.24 g of rough products were obtained.
[0192]b) Manufacture agitating equipment of 4-(2-propenyloxy carbonylamino) benzyl star's picture, In 100-ml 4 mouth flask equipped with the thermometer which can be measured to a reflux condenser and 100 **, 4.24g (20mmol) of 4-(bromomethyl) phenylisocyanates and 25 ml of N.N-dimethylformamide which were obtained by the aforementioned a were put, and it cooled with ice water. After adding three drops of triethylamines, and 1.74 g (30mmol) of 2-propene-1-ol at 7 **, it stirred at the room temperature for 3 hours. Toluene, a tetrahydrofuran, and water were added and extracted to the obtained reaction mixture. The organic layer was washed with water and then it washed with the saturation salt solution, and after drying with anhydrous sodium sulfate, the solvent was distilled off under vacuum pump decompression.
[0193]Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =100:1) is used for a developing solvent for 3.6 g of obtained rough products, Silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined, and the object compound 3.05g of the mark was obtained as white crystals (56% of yield). The melting point of 101-103 **.
[0194] ^{1}H-NMR(delta ppm/CDCl $_3$):4.5 (2H, s), 4.7 (2H, d, J= 5.7 Hz), 5.2-5.4 (2H, m), 5.9-6.1 (1H, m), 6.8 (1H, s), 7.1-7.4 (4H, m).
[0195]Example 24 of reference manufacture - [N-(2-propenyloxy carbonyl)-N-methylamino] Manufacture of a benzyl star's picture 

a) Manufacture agitating equipment of N-methyl-4-tolyl carbamoyl chloride, The die phosgene 1.13g (5.7mmol) was put in 50-ml 4 mouth flask equipped with the thermometer which can be measured to a reflux condenser and 100 **, and 1.33g (11mmol) of N-methyl-4-toluidines and 10 ml of toluene were dropped under stirring at the room temperature. It stirred at 80 ** after the end of dropping for 2 hours. The solvent was distilled off for the reaction mixture under vacuum pump decompression. 2.0 g of obtained rough products were washed by n-hexane, and the mark compound 1.6g was obtained (80% of yield).
[0196]b) Manufacture agitating equipment of N-methyl-4-bromomethyl phenylcarbamoyl chloride, To a reflux condenser and 100 **, 1.6g (8.7mmol) of N-methyl-4-tolyl carbamoyl chloride and 15 ml of carbon tetrachlorides which were obtained by the aforementioned a were put in 50-ml 4 mouth flask equipped with the thermometer which can be measured, and heating stirring was carried out. The mixture of 0.29g (1.7mmol) of 2,2'-azobis (isobutyronitrile) and 1.55 g (8.7mmol) of N-bromosuccinimid was stirred at 75 ** [times / 3] for 3 hours at 70 **. Chloroform and water were added and extracted to the reaction mixture, the chloroform layer was dried with anhydrous sodium sulfate, and the solvent was distilled off under vacuum pump decompression. N-hexane toluene mixture (rate n-hexane of a solvent capacity factor: toluene

=2:1) was used for the developing solvent, silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined 2.3 g of obtained rough products, and the object compound 2.1g of the mark was obtained (92% of yield).

[0197]^{1}H-NMR(delta ppm/$CDCl_3$):3.4 (3H, br. s), 4.5 (2H, s), 7.2-7.5 (4H, m).

[0198]c)4-[N-(2-propenyloxy carbonyl)-N-methylamino] The oily sodium hydride 0.32g (8mmol) and 10 ml of tetrahydrofurans are put 60% in 50-ml 4 mouth flask equipped with the thermometer which can be measured to the manufacture agitating equipment of a benzyl star's picture, a reflux condenser, and 100 **, 0.49 g (8.4mmol) of bottom 2-propene-1-ol of stirring was dropped at 6 **. 2.0 g (7.6mmol) of N-methyl-4-bromomethyl phenylcarbamoyl chloride obtained by the aforementioned b at -7 ** was added after 1-hour stirring at the room temperature. After 2-hour stirring and a reaction mixture were poured out into ice water at the room temperature, and it extracted with toluene, and washed with the saturation salt solution, the toluene layer was dried with anhydrous sodium sulfate, and the solvent was distilled off under vacuum pump decompression. Toluene was used for the developing solvent, silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined 2.1 g of obtained rough products, and the object compound 1.7g of the mark was obtained (79% of yield).

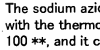
[0199]^{1}H-NMR(delta ppm/$CDCl_3$):3.3 (3H, s), 4.5 (2H, s), 4.6 (2H, d, J= 5.5 Hz), 5.1-5.3 (2H, m), 5.8-6.0 (1H, m), 7.2-7.4 (4H, m).

[0200]Manufacture of an example of reference manufacture 34-(chloromethyl) phenylisocyanate

The sodium azide 0.69g (10.6mmol) and 3 ml of water were put in 50-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **, and it cooled with ice water. 2.0g (10.6mmol) of 4-chloromethyl benzoic acid chloride and the mixed solution of 7 ml of tetrahydrofurans were dropped at 5 **. After 1-hour stirring and a tetrahydrofuran layer were separated at the room temperature, it dried with anhydrous sodium sulfate, and the tetrahydrofuran solution of 4-(chloromethyl) benzoic acid azide was obtained.

[0201]10 ml of toluene was put in 50-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **, and it heated at 80 **. 80 ** was maintained and the tetrahydrofuran solution of the above-mentioned 4-(chloromethyl) benzoic acid azide was dropped. It checked that generating of nitrogen had stopped at 80 ** after 1-hour stirring, and the reaction was ended. The solvent was distilled off for the reaction mixture under vacuum pump decompression, and the mark compound 1.59g was obtained (90% of yield).

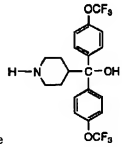
[0202]Manufacture of the example of reference manufacture 44-(bromomethyl) phenylisothiocyanate



6.4g (43mmol) of 4-tolyl isothiocyanates and 50 ml of carbon tetrachlorides were put in and heated to agitating equipment, a reflux condenser, and 100 ** in 200-ml 4 mouth flask equipped with the thermometer which can be measured. The benzoyl peroxide 1.0g (4.1mmol) was put in at 75 **, and then flowing-back stirring was further carried out [succinimid / 7.65 g (43mmol) of / N-bromo/ times / 3] for 1 hour at 75 **. Chloroform and chilled water were added and extracted in the reaction mixture. The chloroform layer was dried with anhydrous sodium sulfate, the solvent was distilled off under vacuum pump decompression, and 9.8 g of rough products were obtained. The rough product was washed by n-hexane and the light yellow crystal thing 8.0g which is a mark compound was obtained (82% of yield). The melting point of 94-99 **.

[0203]^{1}H-NMR(delta ppm/$CDCl_3$):4.5 (2H, s), 7.2-7.4 (4H, m).

[0204]Manufacture of example of reference manufacture 54-[bis(4-trifluoro methoxypheny)

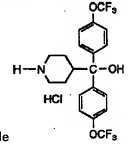


hydroxymethyl] piperidine

a) Manufacture agitating equipment of N-trimethylsilyl isonipecotic acid ethyl ester, In 1l. 4 mouth flask equipped with the thermometer which can be measured to a reflux condenser and 100 **, 25.8 g (0.164 mol) of isonipecotic acid ethyl ester, 360 ml of diethylether, and 17.4 g (0.172 mol) of triethylamine were put in, nitrogen gas replaced the inside of the system of reaction, and it cooled with ice water. 18.9g (0.174 mol) of trimethylsilyl chloride and the mixed solution of 25 ml of diethylether were dropped at 10 **. After stirring at a room temperature for 2 hours, the precipitated crystal was filtered, the filtrate was condensed under vacuum pump decompression, and 34.2 g of rough products were obtained. The mark compound 31.5g was obtained by carrying out distillation under reduced pressure (106-109 ** of boiling points / 4mmHg) of the rough product (84% of yield).

[0205]b) Manufacture agitating equipment of 4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, In 300-ml 4 mouth flask equipped with the thermometer which can be measured to a reflux condenser and 100 **, 0.5 g (2.3mmol) of 4-trifluoro methoxy bromobenzenes, 2.5 ml of tetrahydrofurans, the magnesium 1.4g (57.6mmol), and the iodine 0.01g were put in, and nitrogen gas replaced the inside of the system of reaction. Add 50 ml of tetrahydrofurans after stirring for 10 minutes at 64 **, and at 64 ** 5.3 g (23mmol) of N-trimethylsilyl isonipecotic acid ethyl ester, The mixed solution of 12.9g (53.4mmol) of 4-trifluoro methoxy bromobenzenes and 50 ml of tetrahydrofurans was dropped. The reaction mixture was cooled with ice water after 2-hour flowing-back churning, it flowed into the ammonium chloride solution, and ethyl acetate extracted. After washing with saturated sodium bicarbonate, next washing with a saturation salt solution, it dried with anhydrous sodium sulfate and the mark compound 10.0g was obtained by distilling off a solvent under vacuum pump decompression (100% of yield).

[0206]Manufacture of an example of reference manufacture 64-[bis(4-trifluoro methoxypheny)

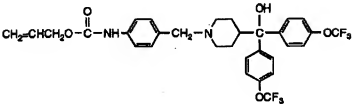


hydroxymethyl] piperidine hydrochloride

In 50-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **, 2.9 g (6.7mmol) of 4-[bis(4-trifluoro methoxypheny) hydroxymethyl] piperidine, 10 ml of diethylether, and 10 ml of n-hexane were put in, and hydrochloric acid gas was introduced for 30 minutes under stirring at the room temperature. The mark compound 2.0g was obtained by filtering the precipitated crystal of the obtained reaction mixture (63% of yield). The melting point of 217-221 ** (decomposition).

[0207]^{1}H-NMR(delta ppm/CD_3C OC D_3):1.6 (2H, d, J= 13.8 Hz), 1.9-2.2 (2H, m), 3.0-3.2 (3H, m), 3.4-3.6 (2H, d, J= 12.6 Hz), 7.2-7.8 (8H, m).

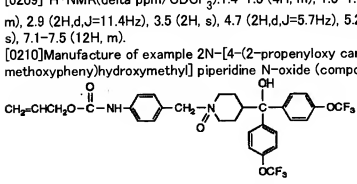
[0208]Manufacture of example 1N-[4-(2-propenyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine (compound number 1)



In 100-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **, 3.0 g (7mmol) of 4-[bis(4-trifluoro methoxypheny) hydroxymethyl] piperidine, 30 ml of chloroform, and 2.0 g (20mmol) of triethylamine were put in and stirred. a room temperature — 4-(2-propenyloxy carbonylamino) benzyl star's picture 1.89g (7mmol) — in addition, it stirred at the room temperature for 2 hours. The obtained reaction mixture was poured out into chilled water, and chloroform extracted. The chloroform layer was washed with the saturation salt solution, it dried with anhydrous sodium sulfate, and the solvent was distilled off under vacuum pump decompression. Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =5:1) was used for the developing solvent, silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined 4.4 g of obtained rough products, and the object compound 2.5g of the mark was obtained as white crystals (57% of yield). The melting point of 148-151 **.

[0209]^{1}H-NMR(delta ppm/$CDCl_3$):1.4-1.5 (4H, m), 1.5-1.7 (1H, br.s), 1.9-2.1 (2H, m), 2.3-2.5 (1H, m), 2.9 (2H,d,J=11.4Hz), 3.5 (2H, s), 4.7 (2H,d,J=5.7Hz), 5.2-5.4 (2H, m), 5.9-6.0 (1H, m), 6.6 (1H, s), 7.1-7.5 (12H, m).

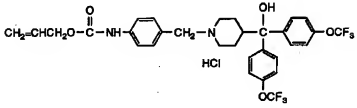
[0210]Manufacture of example 2N-[4-(2-propenyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine N-oxide (compound number 2)



Agitating equipment, To a reflux condenser and 100 **. The thermometer which can be measured. 0.8g (1.3mmol) of N-[4-(2-propenyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine and 10 ml of chloroform were put in and stirred in equipped 30-ml 4 mouth flask. the bottom of the ice water cold, and 7 ** — m-chloroperbenzoic acid 0.32g (1.3mmol) — in addition, it stirred at the room temperature for 2 hours. The obtained reaction mixture was poured out into the saturated sodium bicarbonate solution, and chloroform extracted. The chloroform layer was washed with the saturation salt solution, it dried with anhydrous sodium sulfate, and the solvent was distilled off under vacuum pump decompression. Chloroform methanol mixture (rate chloroform of a solvent capacity factor: methanol =10:1) is used for a developing solvent for 0.8 g of obtained rough products, Silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined, and the object compound 0.7g of the mark was obtained as white crystals (84% of yield). The melting point of 182-183 **.

[0211]^{1}H-NMR(delta ppm/$CDCl_3$-CD_3OD):1.2-1.4 (2H, m), 2.2-2.5 (3H, m), 3.0-3.2 (4H, m), 4.3 (2H, s), 4.7 (2H,d,J=5.7Hz), 5.2-5.4 (2H, m), 5.9-6.1 (1H, m), 7.0-7.6 (12H, m).

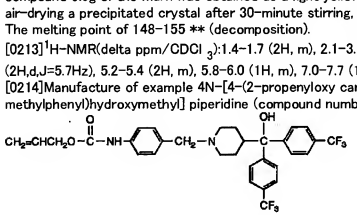
[0212]Example 3N-[4-(2-propenyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny) hydroxymethyl] piperidine Manufacture of a hydrochloride (compound number 3)



Agitating equipment, To a reflux condenser and 100 **. In 50-ml 4 mouth flask equipped with the thermometer which can be measured, 0.8 g (1.3mmol) of N-[4-(2-propenyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, 10 ml of diethylether and 10 ml of n-hexane were put in, and hydrochloric acid gas was introduced for bottom 20 minutes of stirring at the room temperature. Furthermore, after 1-hour stirring and reaction supernatant liquid were decanted at the room temperature, 20 ml of n-hexane was added to residue, and the compound 0.6g of the mark was obtained as a light yellow crystal by isolating by filtration and air-drying a precipitated crystal after 30-minute stirring, at a room temperature (71% of yield). The melting point of 148-155 ** (decomposition).

[0213]^{1}H-NMR(delta ppm/$CDCl_3$):1.4-1.7 (2H, m), 2.1-3.6 (7H, m), 4.0-4.3 (2H, br.s), 4.6 (2H,d,J=5.7Hz), 5.2-5.4 (2H, m), 5.8-6.0 (1H, m), 7.0-7.7 (12H, m), 11.3 (1H, br.s).

[0214]Manufacture of example 4N-[4-(2-propenyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methylphenyl)hydroxymethyl] piperidine (compound number 12)

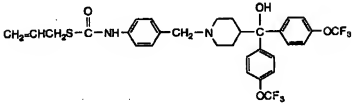


In 100-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **, 0.5 g (8mmol) of 2-propene-1-ol, 10 ml of N.N-dimethylformamide and two drops of diisopropylethylamines were put in, and nitrogen gas replaced the inside of a flask. After adding 0.7 g (4mmol) of 4-(chloromethyl) phenylisocyanates at 2 ** under ice-cooling, it stirred at the room temperature for 1 hour. next, the bottom of the ice water cold -- 5 ** -- the inside of a reaction mixture -- 1.6g (4mmol) of 4-[bis(4-trifluoro methylphenyl)hydroxymethyl] piperidine, and 1.5 g (12mmol) of diisopropylethylamine -- in addition, it stirred at the room temperature further for 3 hours. The obtained reaction mixture was poured out into chilled water, and ethyl acetate was added and extracted.

[0215]The organic layer was dried with anhydrous sodium sulfate after washing in cold water and saturation salt solution washing, and the solvent was distilled off under vacuum pump decompression. Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =5:1) was used for the developing solvent, silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined 2.0 g of obtained rough products, and the object compound 1.7g of the mark was obtained as white crystals (71% of yield). The melting point of 168-170 **.

[0216]^{1}H-NMR(delta ppm/$CDCl_3$):1.4-1.5 (4H, m), 1.5-1.7 (1H, br.s), 1.9-2.1 (2H, m), 2.4-2.5 (1H, m), 2.9 (2H,d,J=11.7Hz), 3.5 (2H, s), 4.7 (2H, d), 5.2-5.4 (2H, m), 5.9-6.1 (1H, m), 6.6 (1H, s), 7.2-7.7 (12H, m).

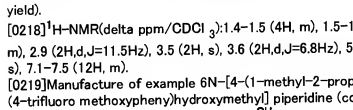
[0217]Manufacture of example 5N-[4-(2-propenylthiocarbonyl amino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine (compound number 40)



0.45 g (6mmol) of 2-propene-1-thiol, 15 ml of tetrahydrofurans, and one drop of triethylamine were put in 50-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **, and it cooled with chilled water. After adding 0.84 g (5mmol) of 4-(chloromethyl) phenylisocyanates at 5 **, it stirred at the room temperature for 3 hours. next, the inside of a reaction mixture -- 2.2g (5mmol) of 4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, and 1.94 g (15mmol) of diisopropylethylamine -- in addition, it stirred at the room temperature further for 2 hours. The obtained reaction mixture was poured out into chilled water, and ethyl acetate was added and extracted. The ethyl acetate layer was washed with the saturation salt solution, it dried with anhydrous sodium sulfate, and the solvent was distilled off under vacuum pump decompression. Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =10:1) is used for a developing solvent for 3.2 g of obtained rough products, Silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined, and the object compound 1.5g of the mark was obtained as a yellow oily matter (47% of yield).

[0218] ^{1}H-NMR(delta ppm/CDCl $_3$):1.4-1.5 (4H, m), 1.5-1.7 (1H, br.s), 1.9-2.1 (2H, m), 2.5-2.7 (1H, m), 2.9 (2H,d,J=11.5Hz), 3.5 (2H, s), 3.6 (2H,d,J=6.8Hz), 5.0-5.3 (2H, m), 5.7-6.0 (1H, m), 7.0 (1H, s), 7.1-7.5 (12H, m).

[0219]Manufacture of example 6N-[4-(1-methyl-2-propenyloxy carbonylamino) benzyl]-4-[bis (4-trifluoro methoxypheny)hydroxymethyl] piperidine (compound number 83)



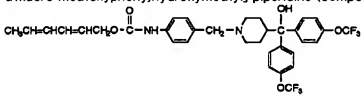
The 3-buten-2-oar 0.43g (6mmol), 10 ml of N.N-dimethylformamide, and two drops of triethylamines were put in 50-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **. After adding 0.5 g (3mmol) of 4-(chloromethyl) phenylisocyanates at a room temperature, it stirred at the room temperature for 1 hour. At a room temperature, in a reaction mixture Next, 1.5 g (3.4mmol) of 4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, 15 ml of tetrahydrofurans, and 1.16 g (9mmol) of diisopropylethylamine -- in addition, it stirred at the room temperature further for 3 hours.

[0220]The obtained reaction mixture was poured out into the saturation salt solution, and ethyl acetate was added and extracted. The organic layer was washed twice with washing in cold water and a saturation salt solution, it dried with anhydrous sodium sulfate, and the solvent was distilled off under vacuum pump decompression. Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =5:1) was used for the developing solvent, silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined 1.9 g of obtained rough products, and the object compound 1.0g of the mark was obtained as a yellow crystal (52% of yield). The melting point of 77-80 **.

[0221] ^{1}H-NMR(delta ppm/CDCl $_3$):1.4 (3H, d, J= 6.4 Hz), 1.4-1.7 (4H, m), 1.9-2.0 (2H, m), 2.2-2.3 (1H, m), 2.9 (2H,d,J=11.7Hz), 3.5 (2H, s), 5.1-5.4 (3H, m), 5.8-6.0 (1H, m), 6.6 (1H, s), 7.1-7.5 (12H, m).

[0222]Manufacture of example 7N-[4-(2,4-hexa dienyl oxy carbonyl amino) benzyl]-4-[bis(4-

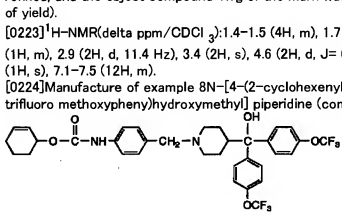
trifluoro methoxypheny)hydroxymethyl] piperidine (compound number 168)



0.6 g (6mmol) of 2,4-HEKISAJI enal, 15 ml of N.N-dimethylformamide, and two drops of triethylamines were put in 50-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **, and it cooled on ice. After adding 0.67 g (4mmol) of 4-(chloromethyl) phenylisocyanates at 10 **, it stirred at the room temperature for 3 hours. the inside of a reaction mixture — 1.7g (4mmol) of 4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, and the potassium carbonate 1.1g (8mmol) — in addition, it stirred at the room temperature further for 3 hours. The obtained reaction mixture was poured out into chilled water, and ethyl acetate was added and extracted. The organic layer was washed with the saturation salt solution, it dried with anhydrous sodium sulfate, and the solvent was distilled off under vacuum pump decompression. Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =5:1) is used for a developing solvent for 2.5 g of obtained rough products, Silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined, and the object compound 1.7g of the mark was obtained as a light yellow oily matter (65% of yield).

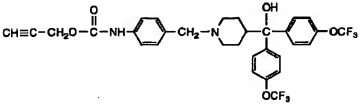
[0223] ^{1}H-NMR(delta ppm/$CDCl_3$):1.4-1.5 (4H, m), 1.7 (3H, d, J= 6.8 Hz), 1.9-2.0 (2H, m), 2.3-2.5 (1H, m), 2.9 (2H, d, 11.4 Hz), 3.4 (2H, s), 4.6 (2H, d, J= 6.6 Hz), 5.6-5.8 (2H, m), 6.0-6.4 (2H, m), 6.7 (1H, s), 7.1-7.5 (12H, m).

[0224]Manufacture of example 8N-[4-(2-cyclohexenyl oxy carbonyl amino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine (compound number 173)



0.6 g (6mmol) of 2-cyclohex Norians, 10 ml of N.N-dimethylformamide, and two drops of pyridine were put in 50-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **, and it cooled on ice. After adding 0.67 g (4mmol) of 4-(chloromethyl) phenylisocyanates at 7 **, it stirred at the room temperature for 1.5 hours. the inside of a reaction mixture — 1.7 g (4mmol) of 4-[bis(4-trifluoro methoxypheny) hydroxymethyl] piperidine -- in addition, it stirred at the room temperature further for 2 hours. The obtained reaction mixture was poured out into chilled water, and the mixture of diethylether and ethyl acetate was added and extracted. The organic layer was washed with the saturated sodium bicarbonate solution, and then it washed with the saturation salt solution, and dried with anhydrous sodium sulfate, and the solvent was distilled off under vacuum pump decompression. Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =5:1) is used for a developing solvent for 2.3 g of obtained rough products, Silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined, and the object compound 1.6g of the mark was obtained as a light yellow oily matter (62% of yield). ^{1}H-NMR(delta ppm/$CDCl_3$):1.4-1.5 (4H, m), 1.6-2.1 (8H, m), 2.3-2.4 (1H, m), 2.9 (2H,d,J=11.3Hz), 3.5 (2H, s), 5.2-5.3 (1H, m), 5.7-5.8 (1H, m), 5.9-6.0 (1H, m), 6.6 (1H, s), 7.1-7.5 (12H, m).

[0225]Manufacture of example 9N-[4-(2-propynyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine (compound number 174)



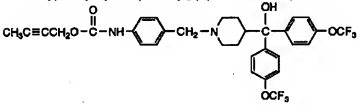
a) Manufacture agitating equipment of 4-(2-propynyloxy carbonylamino) benzyl chloride, In 30-ml 4 mouth flask equipped with the thermometer which can be measured to a reflux condenser and 100 **, 0.50 g (3mmol) of 4-(chloromethyl) phenylisocyanates, 5 ml of chloroform, and two drops of triethylamines were put, and it cooled with ice water. After adding 0.18 g (3.3mmol) of 2-propyn-1-ol at 10 **, it stirred at the room temperature for 2 hours. Chloroform and water were added and extracted to the obtained reaction mixture. After drying an organic layer with anhydrous sodium sulfate, the solvent was distilled off under vacuum pump decompression. The obtained rough product was washed with diisopropyl ether, and the mark compound 0.67g was obtained (100% of yield). The melting point of 121-122 **.

[0226]^{1}H-NMR(delta ppm/$CDCl_3$):2.5 (1H, t, J= 2.4 Hz), 4.6 (2H, s), 4.8 (2H, d, J= 2.4 Hz), 6.7 (1H, bs), 7.3-7.5 (4H, m).

[0227]b) Manufacture agitating equipment of N-[4-(2-propynyloxy carbonylamino) benzyl]-4-[bis (4-trifluoro methoxypheny)hydroxymethyl] piperidine, In 30-ml 4 mouth flask equipped with the thermometer which can be measured to a reflux condenser and 100 **, [Bis(4-trifluoro methoxypheny)hydroxymethyl] 1.31 g (3mmol) of piperidine, 10 ml of dimethyl sulfoxide, 1.16g (9mmol) of diisopropylethylamine, and 0.67 g (3mmol) of 4-(2-propynyloxy carbonylamino) benzyl chloride were put in, and it stirred at the room temperature for 5 hours. Ethyl acetate and water were added and extracted to the obtained reaction mixture. After it rinsed and ranked second and the saturation salt solution washed the organic layer, it dried with anhydrous sodium sulfate and the solvent was distilled off under vacuum pump decompression. Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =5:1) was used for the developing solvent, silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined the obtained rough product, and the object compound 0.60g of the mark was obtained as a light brown oily matter (32% of yield).

[0228]^{1}H-NMR(delta ppm/$CDCl_3$):1.3-1.5 (4H, m), 1.9-2.1 (2H, m), 2.3-2.4 (1H, m), 2.5 (1H, t, J= 2.5 Hz), 2.9 (2H, d, J= 11.6 Hz), 3.5 (2H, s), 4.8 (2H, d, J= 2.5 Hz), 6.7 (1H, s), 7.1-7.5 (12H, m).

[0229]Manufacture of example 10N-[4-(2-butynyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine (compound number 204)

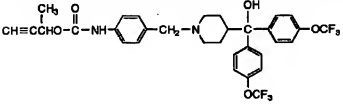


The 2-butine 1-oar 0.42g (6mmol), 15 ml of N.N-dimethylformamide, and two drops of triethylamines were put in 50-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **. After adding 0.5 g (3mmol) of 4-(chloromethyl) phenylisocyanates at a room temperature, it stirred at the room temperature for 1 hour. next, a room temperature — the inside of a reaction mixture — 1.5g (3.4mmol) of 4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, and 1.16 g (9mmol) of diisopropylethylamine — in addition, it stirred at the room temperature further for 3 hours. The obtained reaction mixture was poured out into chilled water, and ethyl acetate was added and extracted. The organic layer was rinsed, it dried with anhydrous sodium sulfate after washing with the saturation salt solution, and the solvent was distilled off under vacuum pump

decompression. Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =5:1) was used for the developing solvent, silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined 2.0 g of obtained rough products, and the object compound 1.0g of the mark was obtained as a yellow crystal (53% of yield). The melting point of 108-110 **.

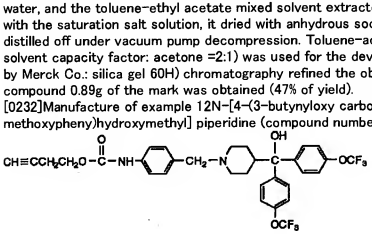
[0230]^{1}H-NMR(delta ppm/$CDCl_3$):1.4-1.5 (4H, m), 1.9 (3H,t,J=2.4Hz), 1.9-2.0 (2H, m), 2.3-2.5 (1H, m), 2.9 (2H,d,J=11.9Hz), 3.5 (2H, s), 4.7 (2H,q,J=2.4Hz), 6.6 (1H, s), 7.1-7.5 (12H, m).

[0231]Manufacture of example 11N-[4-(1-methyl-2-propynyloxy carbonylamino) benzyl]-4-[bis (4-trifluoro methoxypheny)hydroxymethyl] piperidine (compound number 250)



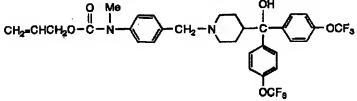
In 30-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **, the 3-butyn-2-oar 0.42g (5.97mmol), 0.50g (2.99mmol) of 4-(chloromethyl) phenylisocyanates and four drops of diisopropylethylamines were put in, it stirred for 30 minutes at 60 **, and 4-(1-methyl-2-propynyloxy carbonylamino) benzyl chloride was obtained. Next, 1.30 g (2.99mmol) of 4in 50-ml mouth [4] flask-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine which equipped the thermometer which can be measured to another agitating equipment, a reflux condenser, and 100 **, 10 ml of dimethyl sulfoxide and 0.58 g (4.48mmol) of diisopropylethylamine were put in, the rough 4-(1-methyl-2-propynyloxy carbonylamino) benzyl chloride obtained at the room temperature in the top was added, and it stirred for 3 hours. The obtained reaction mixture was poured out into chilled water, and the toluene-ethyl acetate mixed solvent extracted. The organic layer was washed with the saturation salt solution, it dried with anhydrous sodium sulfate, and the solvent was distilled off under vacuum pump decompression. Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =2:1) was used for the developing solvent, silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined the obtained rough product, and the object compound 0.89g of the mark was obtained (47% of yield).

[0232]Manufacture of example 12N-[4-(3-butynyloxy carbonylamino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine (compound number 229)



In 50-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **, the 3-butyn-1-oar 0.42g (5.97mmol), 0.50g (2.99mmol) of 4-(chloromethyl) phenylisocyanates and one drop of diisopropylethylamine were put in, and it stirred for 30 minutes at 50 **. Next, 1.30 g (2.99mmol) of 4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine, 10 ml of dimethyl sulfoxide, and 0.77 g (5.97mmol) of diisopropylethylamine were added into the reaction mixture, and it stirred at the room temperature for 3 hours. The obtained reaction mixture was poured out into chilled water, and ethyl acetate extracted twice. The ethyl acetate layer was dried with anhydrous sodium sulfate, and the solvent was distilled off under vacuum pump decompression. Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =2:1) was used for the developing solvent, silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined 1.9 g of obtained rough products, and the object compound 0.98g of the mark was obtained (52% of yield). The melting point of 142-145 **.

[0233]Manufacture of example 13N-[4-(N '- methyl- N'-(2-propenyloxy carbonyl) amino) benzyl]-4-[bis(4-trifluoro methoxypheny)hydroxymethyl] piperidine (compound number 30)



In 50-ml 4 mouth flask equipped with the thermometer which can be measured to agitating equipment, a reflux condenser, and 100 **, 1.35 g (3.1mmol) of 4-[bis(4-trifluoro methoxypheny) hydroxymethyl] piperidine, 10 ml of tetrahydrofurans, and 0.85 g (8.4mmol) of triethylamine are put in, and it is the bottom of stirring, and 4 at a room temperature. - [N-(2-propenyloxy carbonyl)-N-methylamino] The benzyl star's picture 0.8g (2.8mmol) was added, and it stirred at the room temperature for 2 hours. Toluene and water were added and extracted to the reaction mixture, the toluene layer was washed with the saturation salt solution, it dried with anhydrous sodium sulfate, and the solvent was distilled off under vacuum pump decompression. Toluene-acetone mixture (rate toluene of a solvent capacity factor: acetone =5:1) was used for the developing solvent, silica gel (trade name by Merck Co.: silica gel 60H) chromatography refined 1.8 g of obtained rough products, and the object compound 1.0g of the mark was obtained (56% of yield). ^{1}H-NMR(delta ppm/$CDCl_3$):1.4-1.5 (4H, m), 1.6-1.7 (1H, br.s), 1.9-2.1 (2H, m), 2.3-2.5 (1H, m), 2.9 (2H,d,J=11.2Hz), 3.3 (3H, s), 3.5 (2H, s), 4.6 (2H,d,J=5.3Hz), 5.1-5.3 (2H, m), 5.9-6.0 (1H, m), 7.1-7.5 (12H, m).

[0234]The above-mentioned example this invention compound manufactured by the same method as the compounds obtained by 1-13 and these examples is shown in Table 2. The compound number of Table 2 is referred to also in Table 3, 4, and 5.

[0235]The ^{1}H-NMR spectrum data of this invention compound are shown in Table 3. $CDCl_3$ was used for measurement of the ^{1}H-NMR spectrum data of each compound as a solvent, using a tetramethylsilane (TMS) as a standard substance. However, this compound was made suspended for measurement of the compound of the compound number 2 at $CDCl_3$, further, several drops of CD_3OD was added, it was made the transparent solution, and measurement of the ^{1}H-NMR spectrum was presented.

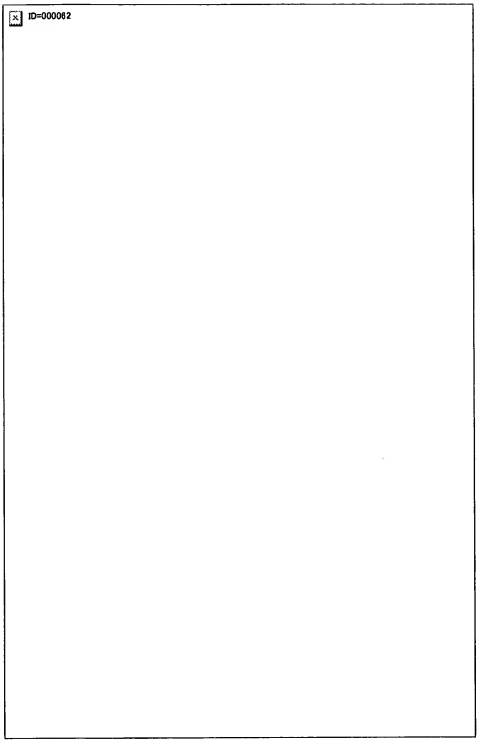
[0236]

【表17】

表2

化合物番号	R_1	R_2	R_3	X	Y	形態	物性
1	CF_3O	H	$CH_2=CHCH_2-$	O	O		mp 148-151℃
2	CF_3O	H	$CH_2=CHCH_2-$	O	O	Nーオキシド	mp 182-183℃
3	CF_3O	H	$CH_2=CHCH_2-$	O	O	塩酸塩	mp 148-155℃ (分解)
12	CF_3	H	$CH_2=CHCH_2-$	O	O		mp 168-170℃
27	tBu	H	$CH_2=CHCH_2-$	O	O		mp 175-179℃
40	CF_3O	H	$CH_2=CHCH_2-$	O	S		オイル
83	CF_3O	H	$CH_2=\overset{CH_3}{\overset{\vert}{C}}HCH-$	O	O		mp 77-80℃
104	CF_3O	H	$CH_2=\overset{CH_3}{\overset{\vert}{C}}CH_2-$	O	O		mp 143-145℃
119	CF_3O	H	$CH_3CH=CHCH_2-$	O	O		mp 156-160℃
140	CF_3O	H	$CH_3\overset{CH_3}{\overset{\vert}{C}}=CHCH_2-$	O	O		mp 132-143℃
129	CF_3O	H	$CH_3O-CH_2CH=CHCH_2-$	O	O		オイル(アメ状)
50	CF_3O	H	$\begin{matrix}Cl\\ \vert\\ Cl-C\end{matrix}=CHCH_2-$	O	O		mp 137-139℃
168	CF_3O	H	$CH_3CH=CHCH=CHCH_2-$	O	O		オイル

173	CF_3O	H		O	O		オイル(アメ状)
174	CF_3O	H	$CH{\equiv}CCH_2-$	O	O		オイル
204	CF_3O	H	$CH_3C{\equiv}CCH_2-$	O	O		mp 108-110℃
250	CF_3O	H	$CH{\equiv}C(CH_3)CH-$	O	O		オイル
229	CF_3O	H	$CH{\equiv}CCH_2CH_2-$	O	O		mp 142-145℃
30	CF_3O	Me	$CH_2{=}CHCH_2-$	O	O		オイル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【表19】

表3(続き)

化合物番号	^{1}H-NMR(δ ppm/CDCl$_3$)
173	1.4~1.5(4H, m), 1.8~2.1(8H, m), 2.3~2.4(1H, m), 2.9(2H, d, J=11.3Hz), 3.5(2H, s), 5.2~5.3(1H, m), 5.7~5.8(1H, m), 5.9~6.0(1H, m), 6.6(1H, s), 7.1~7.5(12H, m)
174	1.3~1.5(4H, m), 1.9~2.1(2H, m), 2.3~2.4(1H, m), 2.5(1H, t, J=2.5Hz), 2.9(2H, d, J=11.6Hz), 3.5(2H, s), 4.8(2H, d, J=2.5Hz), 6.7(1H, s), 7.1~7.5(12H, m)
204	1.4~1.5(4H, m), 1.9(3H, t, J=2.4Hz), 1.9~2.0(2H, m), 2.3~2.5(1H, m), 2.9(2H, d, J=11.9Hz), 3.5(2H, s), 4.7(2H, q, J=2.4Hz), 6.6(1H, s), 7.1~7.5(12H, m)
30	1.4~1.5(4H, m), 1.8~1.7(1H, br.s), 1.9~2.1(2H, m), 2.3~2.5(1H, m), 2.9(2H, d, J=11.2Hz), 3.3(3H, s), 3.5(2H, s), 4.6(2H, d, J=5.3Hz), 5.1~5.3(2H, m), 5.9~6.0(1H, m), 7.1~7.5(12H, m)

The example of pharmaceutical preparation, next the example of pharmaceutical preparation of the compound of general formula (I) by this invention are shown. The "part" in the example of pharmaceutical preparation expresses a weight section.

[0237]As for the additive and addition rate of the example of pharmaceutical preparation which are shown below, it is possible for it not to be limited to the example of these pharmaceutical preparation, and to make it change broadly.

[0238]Example of pharmaceutical preparation 1 About ten copies of emulsion this invention compounds (compound number 1), it is Solvesso 150. It dissolved in 45 copies and 35 copies of

N-methyl-2-pyrrolidone, andX[3005] (made by Toho Chemical Co., Ltd.) 10 copies of SORUPORU were added to this, stirring mixing was carried out, and the emulsion was obtained 10%.

[0239]Example of pharmaceutical preparation 2 20 copies of wettable powder this invention compounds (compound number 12) were added to the inside which mixed two copies of sodium lauryl sulfate, four copies of sodium ligninsulfonate, 20 copies of white carbon, and 54 copies of clay, stirring mixing was carried out by the juice mixer, and wettable powder was obtained 20%.

[0240]Example of pharmaceutical preparation 3 Two copies of sodium dodecylbenzenesulfonate, two copies of carboxymethyl cellulose, two copies of sodium lauryl sulfate, ten copies of bentonites, and 79 copies of clay were added to five copies of granule this invention compounds (compound number 83), and stirring mixing was carried out enough. The water of the adequate amount was added, it stirred further, draught drying was corned and carried out with the granulator, and the granule was obtained 5%.

[0241]Example of pharmaceutical preparation 4 One copy of powder-material this invention compound (compound number 174) was dissolved in two copies of soybean oil, five copies of white carbon, 0.3 copy of acid phosphoric acid isopropyl (PAP), and 91.7 copies of clay were added, stirring mixing was carried out by the juice mixer, and powder material was obtained 1%.

[0242]Example of pharmaceutical preparation 5 20 copies of floor bull agent this invention compounds (compound number 204), and polyoxyethylene alkyl ether, Dialkyl sulfosuccinate sodium and the pro cheating on the fare GXL, respectively Two copies, One copy and 20 copies of water included 0.2 copy were mixed, using dynomill, after wet milling, propylene glycol and xanthan gum were mixed with eight copies and 60 copies of water included 0.32 copy, respectively, and underwater suspension was obtained 20%.

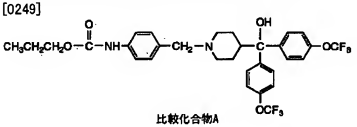
[0243]The example of an examination, next this invention compound show that it is useful as an active principle of an insecticide by the example of an examination.

[0244]Example of an examination After adding acetone, xylene, and a SORUPORU 700HD (made by Toho Chemical Co., Ltd.) solution to each of an insecticidal test this invention compound to one tobacco cutworm, you made it suspended in ion exchange water, and the drug solution (100 ppm) was prepared. It was air-dry after the cabbage folia (8 cm in diameter) was immersed in this drug solution. The folia was put into the plastic cup which covered with the filter paper, the insects scatter of the 3 age larva 10 individual of the tobacco cutworm which shows resistance to a synthetic pyrethroid agent, organophosphorus compounds, the Cava mate agent, and kitchen synthesis inhibitor was carried out, and it covered with the lid which made the small hole, and settled into a 25 ** thermostatic chamber. The mortality of the tobacco cutworm was investigated six days after processing. The test result was shown in Table 4.

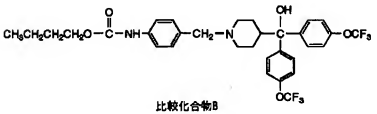
[0245]Example of an examination After adding acetone, xylene, and a SORUPORU 700HD (made by Toho Chemical Co., Ltd.) solution to each of an insecticidal test this invention compound to two cabbage moths, you made it suspended in ion exchange water, and the drug solution (100 ppm) was prepared. It was air-dry after the cabbage folia (8 cm in diameter) was immersed in this drug solution. The folia was put into the plastic petri dish which covered with the filter paper, the insects scatter of the 3 age larva 10 individual of the cabbage moth which shows resistance to a synthetic pyrethroid agent, organophosphorus compounds, the Cava mate agent, and kitchen synthesis inhibitor was carried out, and it covered, and settled into a 25 ** thermostatic chamber. The mortality of the cabbage moth was investigated six days after processing. The test result was shown in Table 4.

[0246]Example of an examination After adding acetone, xylene, and a SORUPORU 700HD (made by Toho Chemical Co., Ltd.) solution to each of an insecticidal test this invention compound to 3 tea HAMAKI, you made it suspended in ion exchange water, and the drug solution (100 ppm) was prepared. It was air-dry after tea leaves (five sheets) were immersed in this drug solution. The leaf was put into the plastic petri dish, the insects scatter of the 3 age larva 10 individual of tea HAMAKI which shows resistance to a synthetic pyrethroid agent, organophosphorus compounds, the Cava mate agent, and kitchen synthesis inhibitor was carried out, and it covered, and settled into a 25 ** thermostatic chamber. The mortality of tea HAMAKI was investigated six days after processing. The test result was shown in Table 4.

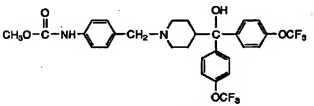
[0247]Example of an examination Four The insect-killing comparative study to the comparison compound A of a statement and the tobacco cutworm of B and C was done on each of an insect-killing comparative study this invention compound, a U.S. Pat. No. 5569664 specification, and the Patent Publication Heisei No. 505080 [nine to] gazette to a tobacco cutworm.
[0248]After adding acetone, xylene, and a SORUPORU 700HD (made by Toho Chemical Co., Ltd.) solution to each of this invention compound, comparison compound A, and B and C, you made it suspended in ion exchange water, and the drug solution (10 ppm) was prepared. It was air-dry after the cabbage folia (8 cm in diameter) was immersed in this drug solution. The folia was put into the plastic cup which covered with the filter paper, the insects scatter of the 3 age larva 10 individual of the tobacco cutworm which shows resistance to a synthetic pyrethroid agent, organophosphorus compounds, the Cava mate agent, and kitchen synthesis inhibitor was carried out, and it covered with the lid which made the small hole, and settled into a 25 ** thermostatic chamber. The mortality of the tobacco cutworm was investigated three days after processing. The test result was shown in Table 5.
[0249]



比較化合物A



比較化合物B



比較化合物C

[0250]
[Effect of the Invention]the new piperidine derivative of this invention has the insect-killing activity which was excellent as shown in the above-mentioned example of an examination, and the immediate effect insect-killing activity which was markedly alike and was excellent in the low dose as compared with the further conventional piperidine compound was accepted.
[0251]

【表 20】

表4

化合物番号	処理濃度 (ppm)	死虫率(%)		
		ハスモンヨトウ	コナガ	チャハマキ
1	100	100	100	100
2	100	100	100	100
3	100	100	100	100
12	100	100	100	100
83	100	100	100	100
174	100	100	100	100
204	100	100	100	100
250	100	100	100	100
229	100	100	100	100
30	100	100	100	100

【表 21】

表5

ハスモンヨトウに対する殺虫比較試験

化合物番号	処理濃度 (ppm)	死虫率(3日後) (%)
1	10	100
2	10	100
3	10	100
12	10	100
83	10	100
174	10	100
204	10	100
250	10	100
229	10	100
比較化合物A	10	0
比較化合物B	10	0
比較化合物C	10	30

[Translation done.]